

DEVELOPMENT OF A MONITOR TO QUANTIFY LEAD-BASED AEROSOLS

By

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This dissertation is dedicated to the memory of my parents.

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KEY TO SYMBOLS

- C - particle concentration
- C_s - slip correction factor
- D - particle diffusion coefficient
- Dp_{50} - impactor cutpoint
- D_t - tube diameter
- g - gravitational constant
- L - tube length
- M - gas molecular weight
- n - number of impaction jets
- P - fractional penetration
- P_s - stack pressure
- Q - volumetric flowrate
- R - bubble radius
- STK - particle Stokes number
- T - gas temperature
- V - gas velocity
- V_b - bubble velocity
- V_p - gas velocity pressure

V_s - particle settling velocity

W - jet width

α_d - coefficient of diffusional deposition

α_i - coefficient of inertial deposition

α_s - coefficient of gravitational deposition

ρ_p - gas density

μ - gas dynamic viscosity

τ - particle relaxation time

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DEVELOPMENT OF A MONITOR TO QUANTIFY LEAD-BASED AEROSOLS

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Manufacture of lead-acid batteries results in the advertent formation of gas-borne particles containing lead. Since the adverse health effects of significant lead exposure are well documented, the battery industry is required to undergo periodic monitoring to demonstrate compliance with existing lead-in-air regulations.

A prototype instrument has been developed to quantify lead-based particulates on a near real-time basis. Aerosol samples can be obtained from high velocity, flowing airstreams as well as from static airstreams. The instrument thus has potential as an emissions monitor, a recirculation air monitor, or a monitor of workplace environments. The prototype instrument contains an air sampling and transport system, a particle collection section, and

a lead analysis section. Proper sequencing of the system's components and archival of the experimental test results are provided by an IBM-compatible computer interfaced with a data acquisition and control system. The prototype monitor may be operated either in attended or automated sampling and analysis mode. The details of the instrument design will be presented along with results of its laboratory and field evaluation.

CHAPTER 1 INTRODUCTION

Background

Industrial manufacturing processes often result in the formation of gas-borne particles (aerosols) containing hazardous metals such as lead, cadmium, and copper. Since the adverse environmental and occupational health effects of these substances are well documented, periodic air monitoring of manufacturing processes involving their use is required to demonstrate compliance with existing federal and local regulations.

The lead acid battery industry, which accounts for 72% of all domestic lead usage (Environmental Protection Agency, 1986), is a potential source of significant lead-based aerosol production. Monitoring of lead emissions in the battery industry typically requires a separate aerosol sampling and analysis technique. First, a representative aerosol sample is obtained from the process using approved sampling techniques. This procedure typically involves aerosol collection through a high efficiency filter to remove particles from the airstream. The collected aerosol sample must then be extracted from the collection substrate and analyzed using an appropriate analytical technique. However, the time delay between sample collection and analysis is typically on the order of

days to weeks. Moreover, improper handling and extraction of the collected sample can result in significant errors in estimation of the total lead concentration. Positive sample bias is a particular problem during measurement of trace lead levels common during evaluation of occupational or recirculation air monitoring. Finally, specially approved equipment, sampling and analysis techniques, and trained personnel are necessary to achieve accurate results. The need existed, therefore, for an instrument capable of providing both aerosol sampling and analysis of lead-based particles on a near real-time basis.

The advantages of an automated air sampling and analysis instrument for lead measurement tests are obvious. Such a device could be used both for occupational and emission measurements and would significantly reduce the cost of each test. Such an instrument would report test results within minutes of the air sampling. Finally, by proper design and construction of the instrument, the sources of measurement error inherent to the current methodology could be significantly reduced. Compliance with existing emission and occupational regulations could be routinely demonstrated. This report details the design, construction, calibration, and field evaluation of such an instrument designed for use by the lead-acid battery industry.

Description of Battery Production Process

The design criteria for the lead-in-air monitor was based on careful review of the various operations involved in the manufacture of lead-acid batteries. Of particular importance was consideration of the concentration, size distribution, and chemical composition of the lead-based aerosols produced at each of the process points. Figure 1-1 is a flow chart of the various operations involved in the battery production. Not shown in the figure are various paths involving the reclaim and reuse of lead scrap which occurs at several of the process points.

Battery manufacture first begins with the production of lead monoxide powder by grinding solid lead metal in the presence of air at elevated temperatures. Lead oxide paste is then produced by combining the lead oxide powder with water and dilute sulfuric acid along with small amounts of proprietary materials.

Battery plate grids are cast from molten lead-based alloys which consist primarily of lead but also contain various trace metals to improve the plate's mechanical properties. Following a specified curing time, the grids are transported to the pasting operation where they are pasted and flash dried prior to being off-loaded from the assembly line. The pasted plates undergo another curing process before use. Plate stacking then occurs which involves alternate stacking of plates with paper separators. The stacked plates are then loaded into plastic cases and the appropriate cells connected together during the cast-on-strap operation. The case cover is then installed, cells filled with sulfuric

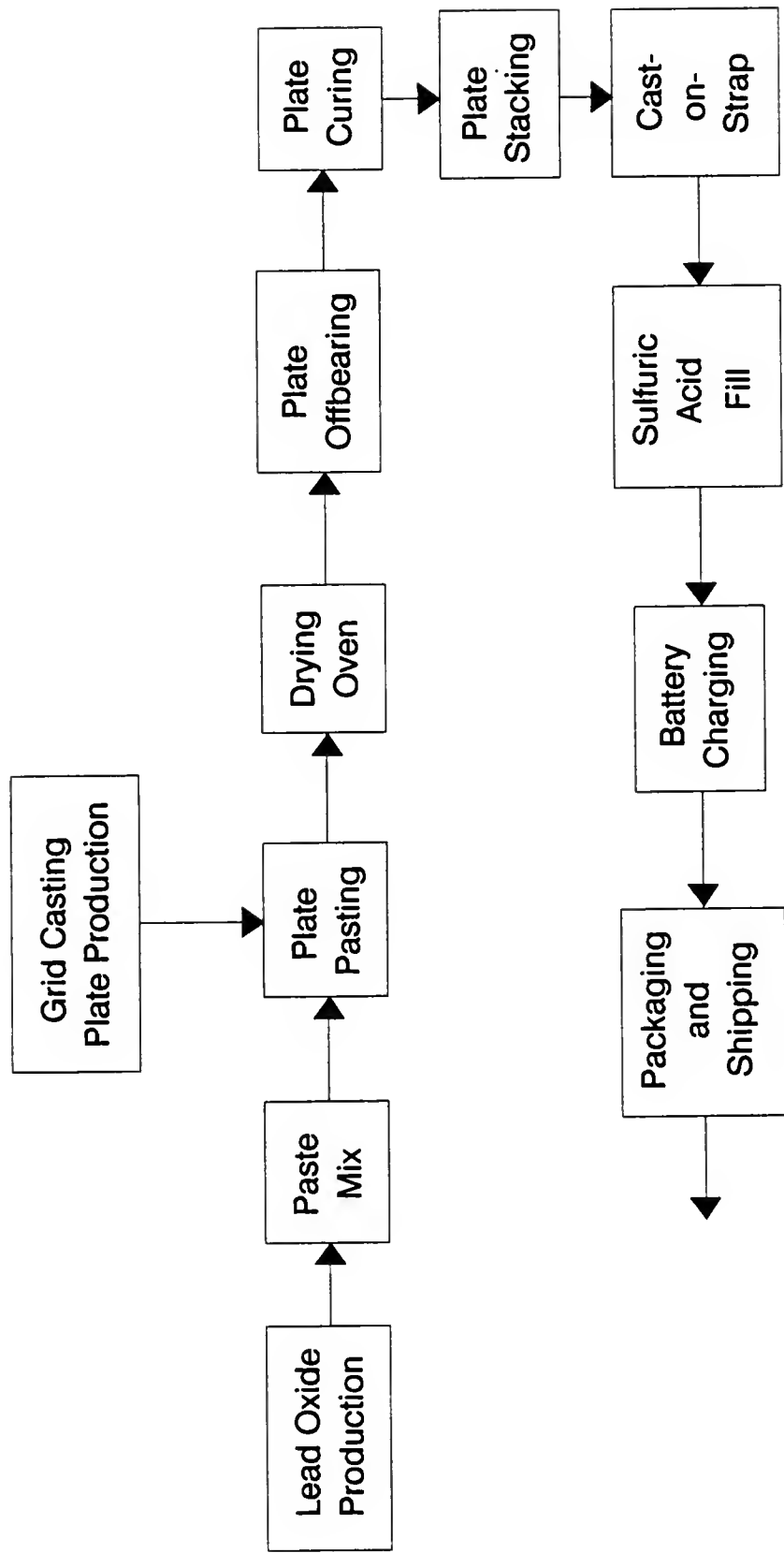


Figure 1-1. Flow chart of lead-acid battery production process.

acid, and the batteries electrically charged prior to the packaging and shipping operation.

The characteristics of the lead-based aerosol produced at each step of the production process depends primarily on the composition of the material handled and the mechanism by which the aerosol is produced. The aerosol produced during the grid casting operation tends to be relatively small in size and may contain only 5% to 20% lead by mass. Lead mass concentrations of aerosol produced at the grid casting operation typically meet allowable emission limits and are therefore ducted to the environment without the need for any control measures.

Conversely, production and handling of lead oxide powder, paste and pasted plates tends to form dispersion-type aerosols relatively large in diameter and contain 95% to 99% lead by mass. Aerosols from these processes also tend to be of much higher lead mass concentrations and are therefore ducted to control equipment (typically baghouses) prior to the airstream's exhaust to the environment.

With the exception of the acid fill, charging and packaging operations, all the steps in the battery production process are sources of elevated lead exposure to the battery plant workers. These processes, therefore, all possess ventilation systems designed to remove the majority of the lead-based aerosols and transport them to the particulate control equipment. Since these airstreams are ultimately exhausted to the environment, an equivalent supply of makeup air

is required to maintain a flow balance. Battery plants in colder climates have incorporated recirculation air systems to reduce heating costs associated with heating the makeup air to plant temperature. This recirculation air is typically obtained from grid pasting, drying, plate offbearing, cast-on-strap, and plate stacking operations and is cleaned through a system of two high efficiency particulate air filters (HEPA) in series with one another. This clean recirculation air is then supplied to various process points as a source of makeup air.

The Occupational Safety and Health Administration has approved use of this type of recirculation air system but requires continuous monitoring of the recirculation air to ensure that the airborne lead content not exceed the regulated 50 micrograms/m³ permissible exposure limit concentration. Experience has shown that if the double HEPA filter system is installed and maintained correctly, concentrations much lower than 5 micrograms/m³ can be routinely obtained.

It is primarily for the purpose of monitoring recirculation air quality that the lead-in-air monitor was developed. Other potential applications to the battery industry will be discussed.

Instrument Design Criteria

The development of a lead-in-air monitor must be based on the sampling and analysis requirements specific to the lead-acid battery industry. First, the instrument must be capable of obtaining representative aerosol samples from

both flowing airstreams (stack or duct sampling) as well as from static airstreams (room sampling). Particle size distribution measurements have shown that efficient particle collection must take place over a fairly wide range of particle sizes.

Accurate quantitation of the particulate lead content is also essential. The analytical technique chosen must be specific for elemental lead and possess minimum physical and chemical interferences from other particulate and gaseous components. Analytical accuracy and repeatability must exist over the measurement range of interest. The analytical technique must possess low blank and background response for the lead measurement.

Sampling should be performed over a 10 to 20 minute time period. Instantaneous measurements are neither technologically feasible nor particularly desirable because variations in lead handling processes and activities result in considerable concentration variability with time. Time averaged measurements, therefore, are more appropriate indicators of average process emissions. In conjunction with a 15 minute analysis time, a 15 minute air sampling period would allow a complete sampling and analysis cycle to be repeated approximately every 30 minutes.

The instrument must also be capable of quantifying lead levels over a wide range of concentrations. These concentrations range from recirculation system alarm levels of 10 micrograms/m³ to levels of greater than 1000 micrograms/m³ for emissions from paste mixing facilities and other three-part operations.

Instrument reliability and ease of use was also a design consideration. Cleaning, maintenance, and external calibration of the instrument should be minimized. The working instrument must be at least semi-automated, fairly portable, and operate on standard 110 VAC, 60 hz power sources.

Finally, it was desired that the instrument's development and construction costs be kept to a minimum. Once a final instrument design had been completed and its capability demonstrated, it was desired that duplication costs be limited to approximately \$10,000.

Summary of Design Approach

Development of an automated sampling instrument for the specific quantitation of lead involved careful consideration of the design criteria outlined in the previous section. Ideally, the developed instrument would satisfy all of the listed criteria although certain design and operational tradeoffs could have been made if necessary. Of primary concern, however, was the instrument's ability to obtain representative particulate samples from particle-laden airstreams and accurately quantify lead levels over a reasonable concentration range.

Design of the instrument can be conveniently divided into three separate sections: the air sampling system, the particulate sampling system, and the lead analysis system. The sampling system must efficiently capture particles over a range of sizes and effectively deliver them to the lead analysis section.

Representative sampling in flowing airstreams requires that isokinetic conditions

be achieved (nozzle velocity equal to freestream velocity) through proper combination of nozzle inlet area and sampling flowrate. It was envisioned that the sampling system contain several nozzles of varying inlet areas in conjunction with a 60 to 90 liter per minute (lpm) capacity sampling pump whose sampling rate could be controlled and monitored by the user. The range of operating flowrates was selected to be 10 to 30 lpm. Following consideration of available particle collection techniques (filtration, electrostatics, etc.), inertial impaction was chosen as the mechanism to be employed in the instrument's particle collection section. Subsequent calibration of the impactor showed that it would collect 95% to 100% of the lead-based particles present in the in-plant processes of interest.

Design of the instrument's analytical section required careful consideration of its specific operating requirements. Of primary concern was the ability to accurately quantify collected lead in amounts which were expected at the various in-plant operations. Considering a 15 minute sampling time and a nominal 15 lpm sampling flowrate, approximately 0.23 m^3 of air would be sampled. For expected concentrations ranging from 10 micrograms/ m^3 to 2000 micrograms/ m^3 at the various in-plant processes, lead deposits of 2.3 micrograms to 460 micrograms would result. If these deposits were placed in liquid solution (50 ml, for example), lead concentrations of 46 micrograms per liter (ppb) to 9 milligrams per liter (ppm) by mass would be expected.

A wide variety of analytical techniques was considered during design of the instrument's analytical section. Most of these techniques were eliminated

from consideration either due to their high cost, level of complexity, lead detection capability, or failure to lend themselves to convenient automation. Ultimately, an electrochemical technique was chosen for the quantitation of lead in liquid solution. A commercially available polarograph was purchased and adapted for automation.

A schematic of the completed instrument is shown in Figure 1-2. Air flow through the system's particle collection section is provided by a 90 lpm capacity air pump with flow controlled and monitored using a commercially available mass flow controller. Deposition of the lead-based particles takes place primarily by inertial impaction into 50 ml of 0.5M nitric acid which is supplied from a 5 liter capacity reservoir. Following a sufficient sampling time, an aliquot of the solution is transferred to a flow-through cell in the polarograph for lead analysis. The lead-in-air concentration can then be calculated from the sampling time, sampling flowrate, solution volume, and the measured liquid lead concentration.

Automated control of the system's components takes place using a data acquisition and control system (Kiethley Instruments, Inc., Model 575) interfaced with an IBM-compatible computer. Computer control not only allows for proper sequencing of the various components but also allows for data reduction and automatic storage and retrieval of the test results. The Keithley 575 data acquisition and control system receives necessary power from the computer and provides both digital and analog input/output capabilities. A Keithley Model AMM1 analog measurement module provides eight separate analog input

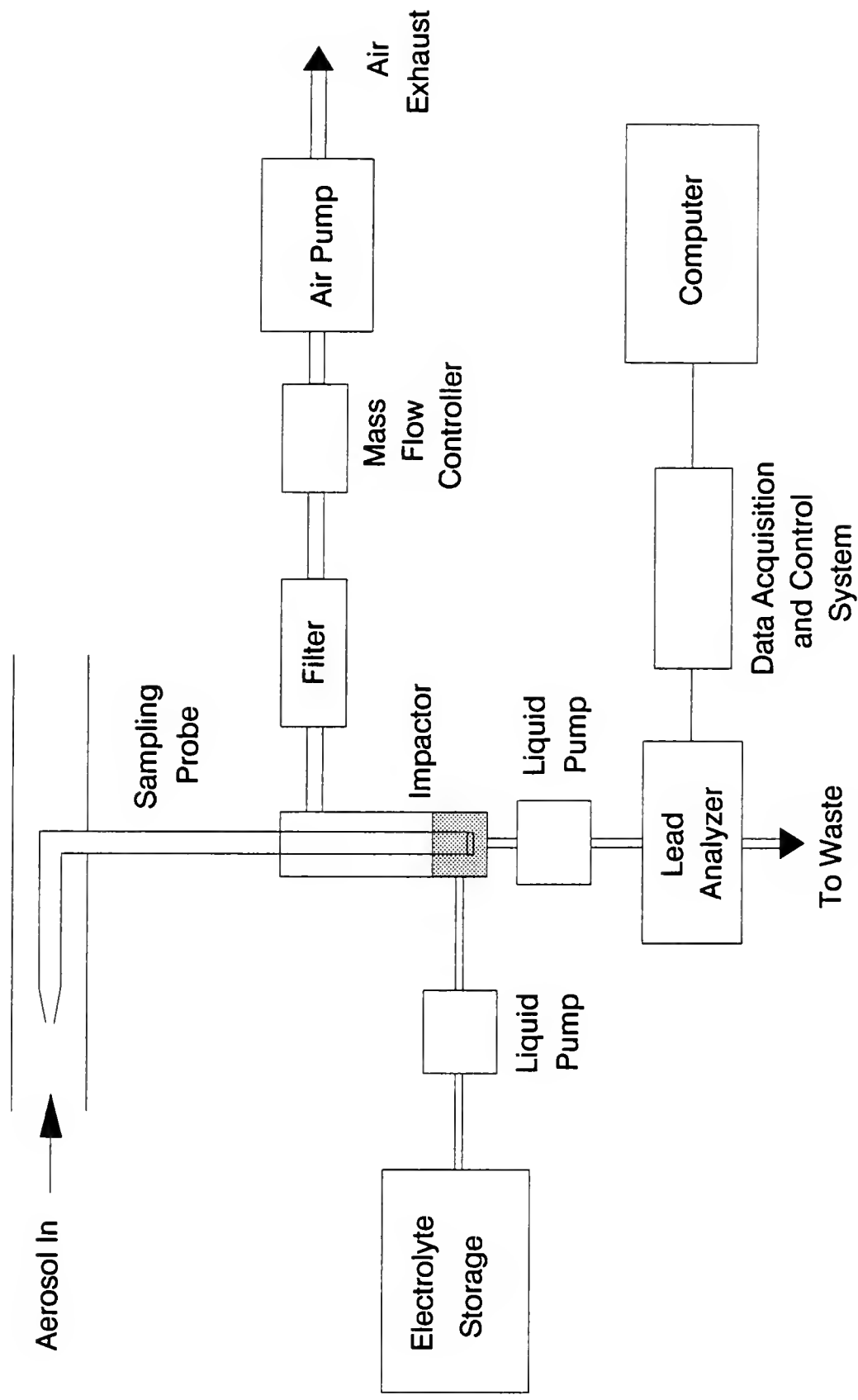


Figure 1-2. Summary of prototype lead-in-air monitor design approach.

channels with a range of $\pm 10V$, an input rate of 50 khz, and 12 bit measurement resolution. The Kiethley 575 contains two analog output channels with similar capabilities. At power up, the Model 575 self-calibrates the analog channels with an accuracy of $\pm 2\%$.

Digital control is provided by 32 digital input/output channels with signals which are transistor-to-transistor (TTL) compatible. A separate 16 channel module (Kiethley, Model PCM3) provides external AC or DC power control using TTL activated solid-state relays.

In the 575 system, the user can access the various module channels using Kiethley KDAC500/I software linked with a GW-BASIC programming environment. Use of programming logic rather than hard-wired logic allowed for design flexibility during development of the prototype system.

CHAPTER 2 LITERATURE REVIEW

Design of the proposed lead-in-air monitor was based on a number of important criteria. The instrument must capture airborne particles in a representative manner and transfer them effectively to the instrument's lead analysis section. The analytical technique chosen must be specific for elemental lead and yield a response that is linearly proportional to mass concentration. Analytical accuracy and repeatability must exist over the measurement range of interest and possess minimal interference from the collection substrate and other particle components.

During the last two decades, considerable progress has been made towards development of direct reading instruments for aerosol measurement. These devices include electrical aerosol analyzers, condensation nuclei counters, aerodynamic particle sizers, optical sizing and counting devices, and piezoelectric and beta gauge based devices. Note that each of these techniques measure a different property of airborne particles and thus do not provide comparable information. Of these devices, only piezoelectric and beta gauge devices provide direct reading of collected aerosol mass. None of these techniques provide information in terms of the chemical composition of the measured aerosol. Typically, devices such as filters, impactors, or electrostatic

collectors are merely used to capture an aerosol for subsequent analysis by a separate instrument.

Conversely, there exist a wide variety of analytical techniques available for chemical quantitative analysis. Typically, these techniques require that the aerosol sample be previously collected in a separate step and manually transferred to the instrument's analytical section. Some sample pretreatment or extraction is often required to obtain reliable test results.

Rather limited success has been achieved in integrating these separate aerosol sampling and analysis techniques in a single, direct reading instrument. Many of these analytical techniques required for trace level analysis are fairly sophisticated and do not adapt well to the development of inexpensive, portable instruments. These sophisticated techniques often require daily calibration and frequent maintenance to ensure accurate test results. The need for automated sample transfer and preparation also complicates the instrument design.

Several tape samplers are available which provide combined aerosol sampling with mass determination. In these automated devices, air flow is normally directed through a porous tape which acts as the collection medium for the airborne particles. This technique thus provides an integrated sample with results time-weighted for the sampling period. In the GCA Model RDM301 sampler, particle matter is deposited by impaction onto a mylar film coated with an adhesive (Lioy, 1983). At the end of the sampling period, the collected material is quantified by beta attenuation. For low energy beta radiation, the

attenuation is a function of mass density of the absorber and is largely independent of chemical composition or the particle's physical properties. Spagnola (1989) developed an automated sampling instrument based on this analytical principle. The instrument uses electromechanical devices for automatic positioning of membrane filters in the sample holder. At the end of the sampling period, the filter is analyzed and returned to its original position in the sample holder. Application of beta attenuation has also been applied to a dichotomous sampler developed by Macius and Husar (1976).

A paper tape sampler was also modified to provide lead analysis by x-ray fluorescence (Smith et al., 1986). The system samples air at 0 to 10 lpm and irradiates the collected aerosol mass with a 10 mCi cadmium 109 radioisotope. A NaI scintillator in conjunction with a multi-channel analyzer provides counts versus x-ray energy for the resulting x-rays. The count corresponding to the x-ray energy characteristic of lead provides a measure of the collected lead mass. The authors note that the presence of arsenic and zinc positively interfere with the lead measurement.

Automated mass monitors have also been developed based on the concept of piezoelectric sensing of changes in aerosol mass. The TSI Inc. Model 5000 deposits particles onto a piezoelectric crystal by electrostatic precipitation. The rate of change of the crystal's output frequency is directly proportional to the deposited mass concentration. Recently, this mass sensing technique was applied to the development of an instrument which uses a low-frequency tapered element coupled to a filter collector (Wang, 1985).

Successful measurement of aerosol composition "in-situ" has been limited and has generally not evolved beyond the laboratory. Arnold and Folan (1987) devised an instrument which levitates a single airborne particle within an electrostatic field and irradiates the particle with an infrared beam. The instrument optically detects changes in particle size caused by evaporation of the particle due to the heat induced by the infrared absorption. The measured rate of change is proportional to aerosol mass. The instrument is capable of detecting picogram quantities of material for both organic and inorganic functional groups such as sulfate and carbonate (Allen and Palen, 1989).

The majority of analytical techniques which have been adapted for trace aerosol chemical analysis are available only in an "off-line" mode and do not adapt well to field use. Martinsson and Hansson (1988) have used particle induced x-ray emission (PIXE) for the multielement analysis of aerosol previously deposited onto thin aluminum foils. Deposits were stepwise heated to several hundred degrees centigrade by applying an electric current through the sample. An ion beam impinged on the sample during the process. Low atomic weight element information (such as oxygen and carbon) was obtained by particle elastic scattering analysis while elements with atomic numbers greater than 14 were determined by PIXE analysis. The chemical composition of the sample was inferred from noted vaporization temperatures and stoichiometry. Laboratory tests found the technique useful for quantitation of ammonium chloride, ammonium nitrate, and ammonium sulfate.

Xhoffer et al. (1989) used a transmission electron microscope to analyze laboratory generated aerosols deposited onto carbon coated electron microscope grids. The analytical technique of electron energy loss spectroscopy was used to observe the change in kinetic energy as the electron passes through the sample. This technique is generally useful only for low atomic weight elements. The authors also noted that high background signals can be associated with the substrate and that rapid deterioration of environmental samples can occur. The technique was also found to possess limited quantitative application since the particle's absorption of the incident electron beam was a function of particle size.

Electron spectroscopy for chemical analysis (ESCA) was investigated by Muller et al. (1987) as a means of characterizing sub-micrometer sodium sulfate aerosol collected on various filter media. This technique used incident x-rays to excite photoemission of core level electrons from the sample. The measured energy of the emitted photoelectrons is characteristic of the emitting element's oxidation state. ESCA provides only surface analysis and is thus of limited quantitative analysis. ESCA signals can also be difficult to interpret quantitatively. Incorporating a similar surface analytical technique, Fletcher (1983) used a laser microprobe to vaporize and ionize a collected sample for analysis by a time-of-flight mass spectrometer. Reported limitations of this technique were similar to those experienced by Muller et al. using ESCA analysis.

Neutron activation analysis (NAA) has been employed to provide elemental information of collected aerosol samples. In this technique, the sample is irradiated with neutrons to produce radionuclides. The activities of the radionuclide products are then measured and related to the quantities of the elements present in the sample. Podzimek and Wojnar (1990) used NAA to quantify masses of laboratory generated titanium oxide particles deposited on polyester filters. In a related study, Ondov et al. (1990) used NAA to quantify 45 elements in rural sub-micrometer ambient aerosols collected by micro-orifice impaction. Although this technique was found to be generally useful, the substrate blank precluded analysis of several elements of interest.

X-ray powder diffraction analysis of collected aerosol deposits was reported by Tanninen et al. (1985). Powder diffraction allows for direct determination of crystalline compounds. The authors used the diffraction technique to analyze fumes generated by metal arc welding of steel. The main analytical difficulty encountered was low instrument output intensity due to the small particles analyzed in conjunction with the small quantity of material present.

Both conventional flame and graphite furnace atomic absorption spectroscopy have been widely used for the analysis of collected aerosol samples. Davidson (1979) used graphite furnace techniques to characterize size fractionated aerosol samples obtained from three national parks. Attempts to incorporate atomic absorption analysis in an automated monitor have met with limited success (Smith et al., 1986).

CHAPTER 3 DESIGN AND CALIBRATION OF THE PARTICLE COLLECTION SECTION

Design Theory

Accurate quantitation of lead-in-air concentrations requires efficient capture of lead-based particles. If an aerosol was only collected with 10% efficiency, the resulting lead analysis would obviously greatly underestimate the actual lead-in-air concentration. This would, in turn, result in an underestimation of the lead exposure hazard. This section describes the design and calibration of a device capable of sampling and capturing lead-based particles with the necessary high efficiency to ensure accurate test results.

There currently exist a variety of aerosol samplers and collectors which offer a basis for the collector design. These devices include inertial devices (impactors and cyclones), electrostatic precipitators, thermal precipitators, diffusion devices, and filtration devices. Each of these devices relies on a particular property of airborne particles to remove the particles from the airstream in which they are suspended.

Fundamental to the design of any particulate collection section is understanding the nature of the aerosol to be measured. In predicting individual particle properties (settling velocity, diffusional behavior, etc) the most

important parameter is that of particle size. Since industrial aerosols are always a collection of particles of varying sizes, it is understanding of the aerosol's size distribution which is of importance.

Figure 3-1 presents particle size distributions measured at a battery plant in the southeastern United States. The specific sampling locations represent sources of aerosols which supply recirculation air. It is believed, therefore, this size distribution can be assumed to be fairly representative of aerosols which exist in recirculation air systems. Plotted in the figure for the various process points are cumulative lead mass distributions versus aerodynamic particle diameter as measured using a University of Washington cascade impactor. The aerodynamic diameter is defined as the diameter of a spherical particle of unit density which has the same settling velocity as the particle under consideration. Use of an equivalent aerodynamic diameter accounts for the particle's size, shape, and density.

Inspection of Figure 3-1 reveals that these measured size distributions are fairly large with half of the lead mass associated with particles greater than 10 micrometers aerodynamic diameter. These results agree well with similar measurements by Hodgkins et al. (1990) from two different battery manufacturing plants. Note that only 1% to 5% of the lead mass is associated with particles less than one micrometer aerodynamic diameter. A device capable of collecting all particles greater than one micrometer, therefore, would have an overall mass collection efficiency on this aerosol of 95% to 99%.

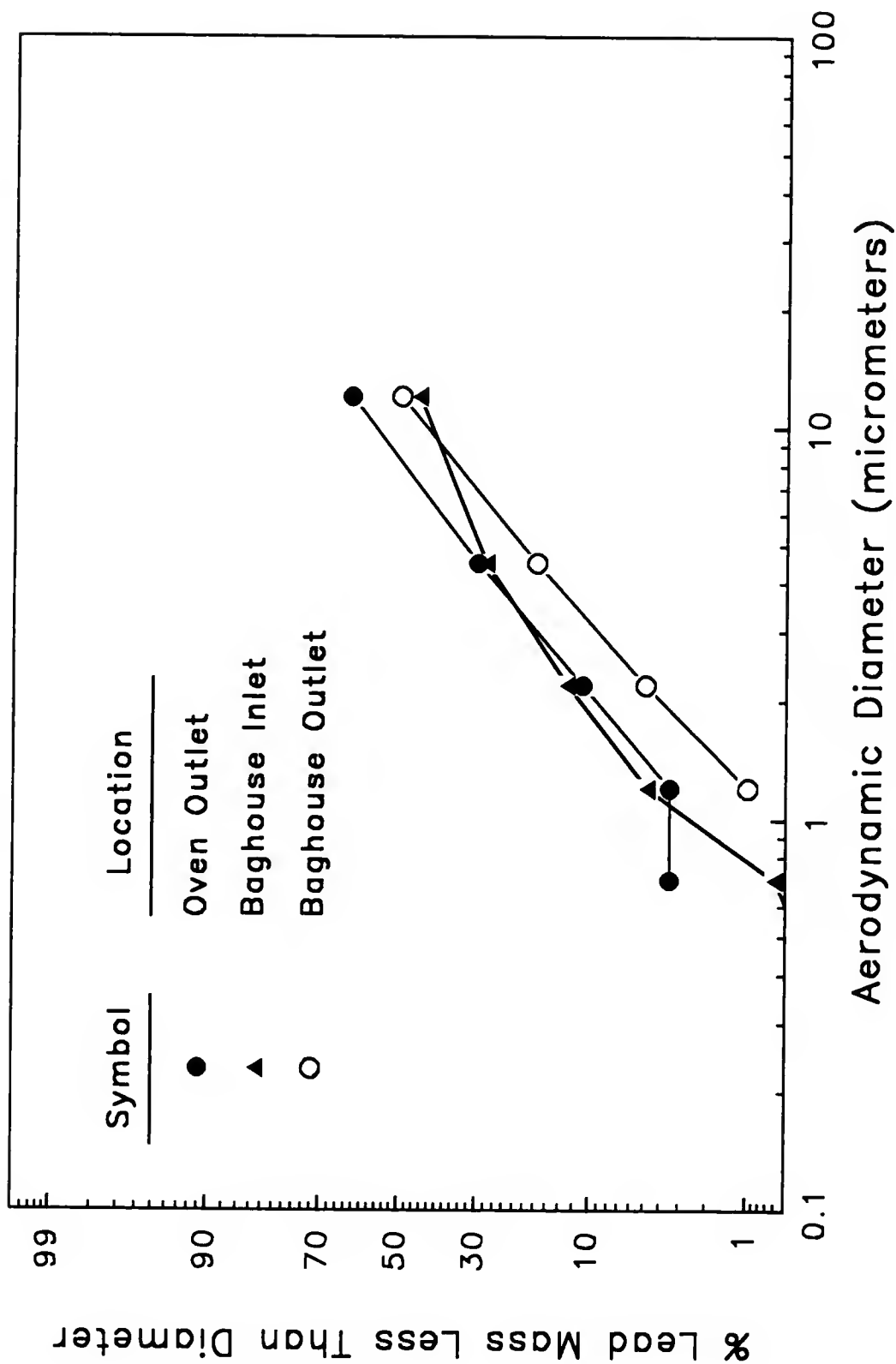


Figure 3-1. Measured particle size distributions at a battery plant.

Based on a review of these distributions, both thermal and diffusion type collectors were eliminated as candidates for the collector design. Although these type collectors can capture submicron aerosols with high efficiency, their capture efficiencies for particles greater than one micrometer are quite low (Hinds, 1982).

Filtration probably represents the most common form of aerosol collection in air sampling. Unlike thermal and diffusion type devices, samplers which rely on filtration mechanisms possess high collection efficiencies over a wide range of particle sizes. Aerosols encountered in the battery industry could be expected to be removed by filtration with virtually 100% efficiency. Despite these advantages, however, filtration was eliminated from design consideration primarily for logistic reasons. Due to high particle-substrate attractive forces, particles collected on high efficiency filters are extremely difficult to remove efficiently even if wet chemical techniques are employed. Wetting of either fibrous or porous membrane filters dramatically increases its pressure drop making the filter difficult to reuse. The alternative to filter reuse is to replace it after each sampling test. Automated replacement of the filter would have involved design and cost complications which were considered to be unacceptably high.

Electrostatic collection was also given consideration during the instrument's design phase. In these devices, collection relies on the electrostatic forces which can be exerted on charged particles in an applied

electric field. Similar to filtration samplers, electrostatic samplers possess high collection efficiencies over a wide range of particle sizes. Use of electrostatic collection would have involved use of a point-to-plane precipitator for deposition of airborne particles to a liquid surface similar to the design approach adopted during the design of the LEAP sampler (Andersen Samplers, Inc.).

This design approach was initially investigated during the instrument development but was ultimately discarded for two primary reasons. First, preliminary laboratory tests with a design prototype showed that the flow of electrical current through the electrolyte resulted in the inadvertent plating out of previously collected lead ions from solution. Loss of collected lead in solution would have resulted in underestimation of the total lead-in-air concentration and thus an underestimation of the lead exposure hazard. Use of electrostatics was also eliminated due its greater complexity and anticipated maintenance requirements in the corrosive nitric acid atmosphere of the collection region.

Ultimately, the use of inertial impaction was adopted for the final instrument design. Inertial impactors have been widely used for aerosol collection and sizing and can efficiently capture particles greater than one micrometer diameter. Inertial impactors are relatively simple in construction, contain no moving parts or electrical components, and require little maintenance.

Impactors rely on the high inertial forces which can be established on particles in a high velocity airstream. In a typical impactor stage, particle-laden

gas is accelerated through one or more nozzles and directed toward a solid surface which forces the airstream to change its flow direction. Due to their inertia, however, particles will be unable to exactly follow their respective streamlines and will deviate from the airstream by a distance which is a function of the particle's aerodynamic diameter and the jet velocity. Particles of sufficient inertia will deviate a sufficient distance to strike the collection surface and be removed from the airstream. Conventional impactor stages with high jet velocities can remove particles of 0.3 micrometers aerodynamic diameter and larger.

Impactor performance is typically described by its Dp_{50} cutpoint which is the particle size collected with 50% efficiency. Particles larger than the cutpoint diameter are collected with much higher efficiency. Extensive theoretical and practical impactor research has shown that the cutpoint can be predicted with some confidence by

$$Dp_{50} \sqrt{C_s} = \sqrt{\frac{9 \pi \mu n W^3}{4 \rho_p Q}} \sqrt{STK} \quad (3-1)$$

where C_s is the Cunningham's slip correction factor determined from the particle size and the fluid properties, μ is the gas dynamic viscosity, n is the number of jets, W is the jet width, ρ_p is the particle density, and Q is the volumetric flowrate. The Stokes value (STK) can be predicted from knowledge

of the jet Reynolds number, ratio of jet width to jet to plate distance, and ratio of jet throat length to jet width.

Based on these accepted design criteria, an inertial collector was designed and constructed for use as the instrument collection section.

Figure 3-2 is a schematic of this device showing key components. Complete detail drawings of each of the unit's components are presented in Appendix A.

In most inertial impactors, the impaction jet is directed onto a preweighed stationary surface previously coated with a viscous, nonvolatile grease to minimize particle bounce and losses due to blowoff. Following the sampling run, the impaction surface is reweighed to quantify the mass of the collected aerosol deposit. If chemical analysis is to be performed, the aerosol must be extracted from the grease in an efficient and representative manner.

The collector developed for the lead-in-air monitor does not impact particles onto a solid surface but into 50 ml of 0.5 M HNO_3 . Use of the liquid impaction substrate eliminates the need for greased substrates and provides for direct transfer of the particles from the gaseous phase to the liquid phase. High velocity impaction also provides for the liquid turbulence necessary for efficient particle dissolution so that aqueous lead ions are formed. High liquid turbulence also tends to homogenize the liquid sample so that only an aliquot of the sample need be analyzed for accurate lead determination.

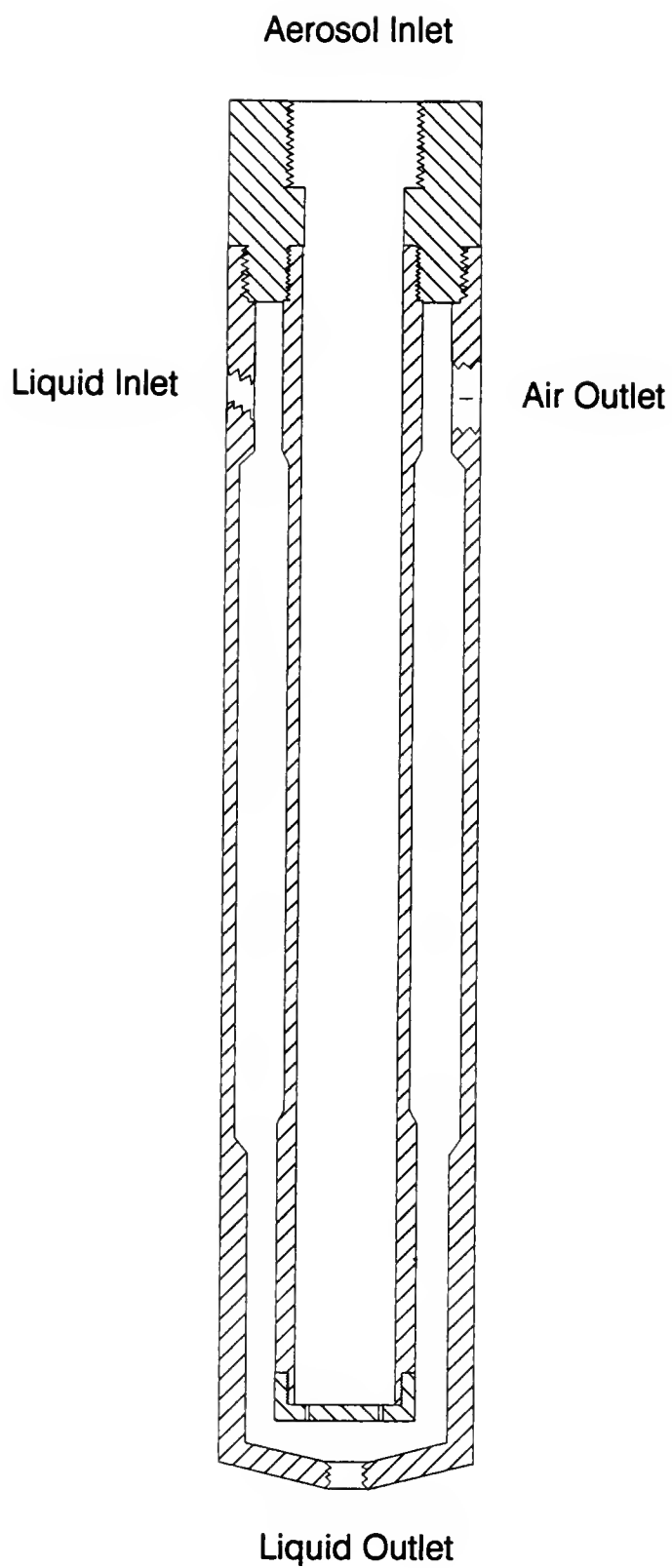


Figure 3-2. Schematic of single-stage impactor used for particle collection.

During the sampling run, particle laden air enters the impactor inlet at a flowrate of 10 to 30 lpm (depending on the sampling situation) and is accelerated through a single stage impactor. As shown in Appendix A, the impaction stage contains 20 circular jets of 0.026 inch diameter. Following its impingement into the liquid, the airstream then enters an expansion section which reduces the upward air velocity and thus reduces inadvertent liquid carry over. Use of a small plug of glass fiber filter at the air outlet has shown to further minimize the liquid carry over. At the conclusion of the air sampling period, a liquid outlet provides for transfer of the solution to the instrument's lead analysis section.

Prediction of Impactor Performance

Inspection of Equation 3-1 allows for the generalized prediction of impactor performance as a function of sampling air flowrate and the critical impactor dimensions. Table 3-1 presents a summary of these calculations assuming standard temperature and pressure conditions for air. Also presented are actual measured stage pressure drops as a function of the sampling flowrate. Calculated cutpoints at flowrates of 10, 20, and 30 lpm are 0.92, 0.63, and 0.42 micrometers, respectively.

It should be emphasized that these calculations are based primarily on theoretical and experimental evaluations of impaction onto a solid surface. However, immersion of an impaction stage and impingement of an airstream

Table 3-1
Predicted Operating Characteristics of Teflon Impactor

Flow (lpm)	Velocity (cm/sec)	Reynolds No.	$(STK)^{1/2}$	$D_p (C)^{1/2}$ (micrometers)	D_p (micrometers)	Measured ΔP (mm Hg)
10	2440	1070	0.48	1.00	0.92	10
20	4880	2150	0.48	0.71	0.63	24
30	7310	3220	0.48	0.50	0.42	46

into a liquid represents an entirely different flow situation. While impaction is certainly the most dominant collection mechanism involved, the entire process may more correctly be described as a bubbling phenomenon.

Unlike the extensive studies related to conventional inertial impaction, there has been relatively limited research devoted to the absorption of aerosols by bubbling. As a simplified model, some researchers have considered particle deposition in air bubbles which freely rise through a stationary liquid. As a further simplification, these studies only consider the motion of bubbles less than 0.2 cm diameter which have been found to be approximately spherical in shape. Larger bubbles become flattened as they rise through the liquid and pulsations in the bubble shape can occur.

Deposition of particles with a buoyantly rising bubble results primarily due to the relative motion of the bubble with respect to the liquid. Due to the shear force which exists at the liquid-gas interface, a gas circulation within the bubble is established and maintained. As a result of this circulation pattern, large particles may be removed by inertial impaction to the liquid-gas interface. Gravitational settling of large particles can also occur as well as removal of submicron particles by molecular diffusion.

The first analytical and experimental treatment of this particle absorption phenomenon within a freely rising bubble was performed by Davies and Taylor (1950). This theory was extended by Fuchs (1964), who developed coefficients of absorption (deposition efficiency per unit cm of bubble rise) for the separate

absorption mechanisms of inertial impaction, sedimentation, and diffusion.

Later, related experimental studies by Melandrie and Prodi (1971) confirmed the validity of this approach.

The coefficient of absorption due to inertial deposition, α_i , is related to the bubbles upward velocity, V_b , the particle's relaxation time, τ , and the radius of the bubble, R , by

$$\alpha_i = \frac{9 V_b \tau}{2 R^2} \quad (3-2)$$

Because particle relaxation time increases with the square of the particle diameter, this inertial mechanism is dominant for large particles.

Similarly, the absorption coefficient due to particle sedimentation, α_s , is expressed as

$$\alpha_s = \frac{3 g \tau}{4 R V_b} \quad (3-3)$$

For most bubble sizes of interest, inertial deposition is an order of magnitude greater than that of sedimentation.

Finally, diffusional deposition efficiency per unit path length can be calculated by accounting for the particle's diffusion coefficient, D

$$\alpha_d = 1.8 \sqrt{\frac{D}{V_b R^3}} \quad (3-4)$$

Large bubbles ($R > 1$ cm) rising in a liquid tend to flatten and pulsate such that their rate of rise is difficult to predict. However, smaller bubbles can be assumed to be spherical and their upward velocity can be predicted by an empirical relationship developed by Melandrie and Prodi (1971)

$$V_b = 3.05 \sqrt{R} \quad (3-5)$$

where the dimensions of V_b and R are m/sec and m, respectively.

For a known bubble size and particle diameter, the overall change in particle concentration (C) per unit length of bubble rise can be calculated by

$$\frac{dC}{dx} = - (\alpha_i + \alpha_s + \alpha_d) C \quad (3-6)$$

For example, Jonas and Schulz (1991) used this relationship to calculate an overall collection efficiency of 98.8% of one micrometer radius iron oxide particles within 0.5 cm radius bubbles buoyantly rising through one meter of liquid.

Inspection of the absorption equation reveals that for a given particle size, decreasing the bubble radius results in an increase in absorption efficiency. The equation also indicates that for a given bubble size, the dominant collection mechanism depends strongly on particle size. For large particles (greater than 0.3 micrometers radius) which possess large relaxation

times, inertial and sedimentation forces dominate while diffusive absorption is negligible. Conversely, particles less than 0.05 micrometers radius are removed almost entirely by molecular diffusion. Thus, regardless of bubble diameter, there exists an absorption minimum which exists for particles of approximately 0.2 to 0.4 micrometer diameter. The actual magnitude of the minimum depends primarily on bubble radius.

Use of these theoretical considerations to exactly predict the performance of the lead-in-air monitor's impactor proved to be difficult. This simplified approach assumes bubbles of known size rising through a stable liquid of constant height. Experimental verification of the theory by other researchers has been limited and performed at relatively low flow rates using uniform sized bubbles. However, the dynamics within the teflon impactor represent a vastly different flow situation. Temporary replacement of the impactor's opaque housing with a glass enclosure of similar dimensions allowed for the direct observation of the bubbling phenomenon. This observation revealed the limitations of the simplified theory to this particular collector. First, it was evident that the bubbles produced were not uniform in size but existed over a wide range of sizes. To apply the absorption theory to a distribution of bubble sizes, the distribution itself must be known and the collection efficiency equation integrated over the bubble size range. At these high flowrates, it was also evident that the bubbles themselves experienced considerable oscillation and occasional breakup due to the high liquid turbulence. Finally, it was

observed that the liquid itself underwent considerable turbulence which changes the nature of the liquid-bubble interface and results in uncertainties in predicting the path rise of the individual bubbles.

Due to this lack of reliable design theory, no exact predictions of the impactor's actual performance were made. Preliminary review of conventional impactor design theory and bubbling theory were used only as a basis for the impactor design. For accurate evaluation of the impactor's actual performance, extensive experimental efficiency tests were conducted with the impactor in the laboratory.

Calibration of the Particle Collection Section

In evaluating the performance of the particle collector, it was understood that the collection efficiency would depend on the unit's critical dimensions, liquid solution volume, particle diameter, and the sampling flowrate. Since the impactor's dimensions and the solution volume were fixed by design, the particle size and sampling flowrate were normally the only two parameters varied during the collection efficiency tests.

The particle collection characteristics of the impactor were evaluated in the laboratory using monodisperse calibration aerosols of solid, spherical ammonium fluorescein. These calibration aerosols were produced by two separate generation systems depending upon the size of the particle required.

For the generation of calibration aerosols 0.5 micrometers aerodynamic diameter and larger, a Thermal Systems Inc. Model 3050 vibrating orifice

aerosol generator (VOAG) was used. A schematic of the complete generation and sampling system is presented in Figure 3-3. The VOAG operates on the principle of the controlled mechanical breakup of a liquid jet into uniform droplets of predictable size. Periodic breakup of the liquid jet was achieved by application of an AC signal at a known frequency to a piezoelectric crystal which housed the liquid orifice. Since the liquid consisted of a known quantity of a non-volatile solute dissolved in a volatile solvent, the generated droplets dried to form uniform particles of predictable size. For the calibration of the impactor, the liquid solution was prepared by dissolving a fluorescein powder in aqueous ammonia. After their production, the droplets dried to form spherical particles of solid ammonium fluorescein. The particles were then mixed with clean dilution air and charge neutralized by exposure to a ^{85}Kr radioactive source. The particles were then mixed with additional dilution air and sampled by the impactor for the calibration tests. An optical microscope was used to verify the size and quality of the generated particles collected on a fibrous or membrane filter before each test series. Figure 3-4 is a scanning electron photomicrograph of a monodisperse aerosol generated by this technique.

For calculating the particle's aerodynamic diameter from its physical diameter, an ammonium fluorescein particle density of 1.35 g/cm^3 was assumed. This value was based on density measurements by Stober and Flachsbart (1973), John and Wall (1983), Vanderpool and Rubow (1988), and Chen and Crow (1986).

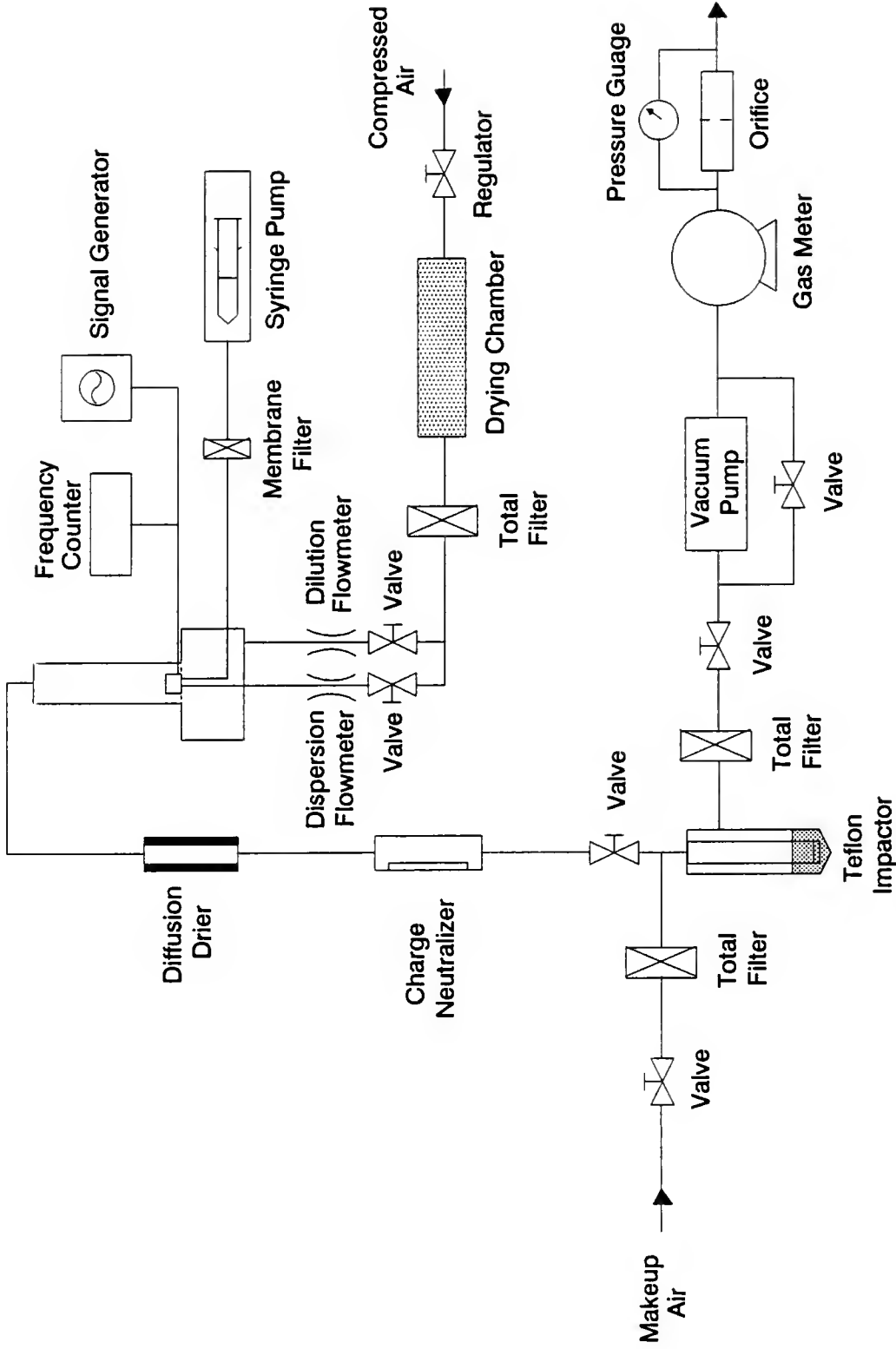


Figure 3-3. Schematic of generation system used for production of calibration aerosols greater than 0.5 micrometers aerodynamic diameter.

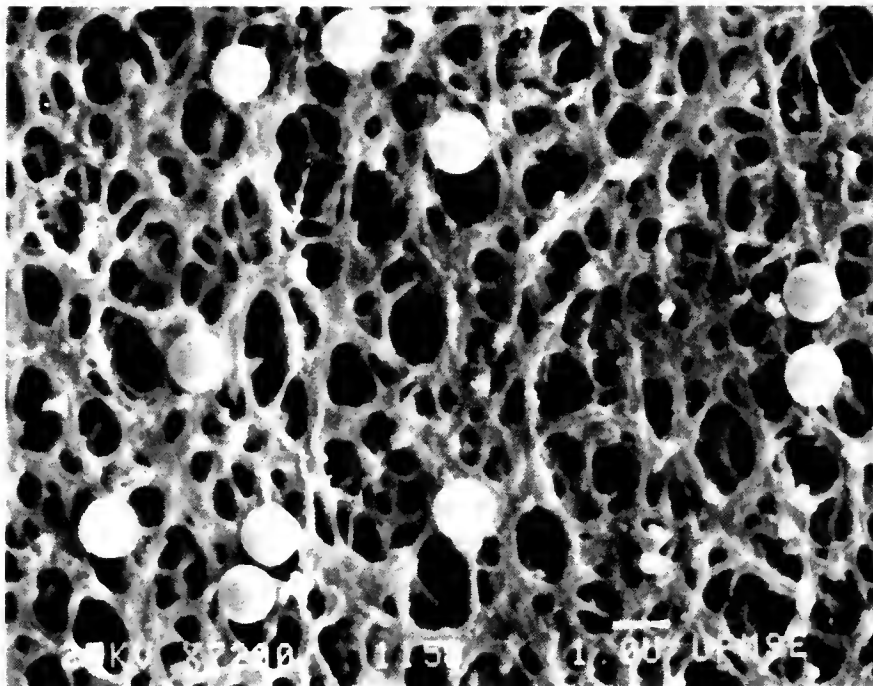


Figure 3-4. Scanning electron photomicrograph of ammonium fluorescein calibration aerosols produced by VOAG.

Calibration aerosols less than 0.5 micrometers aerodynamic diameter were generated using a Thermal Systems Inc. Model 3071 electrostatic classifier. Figure 3-5 is a schematic of this generation setup. Polydisperse droplets were first generated by air nebulization of fluorescein solutions through a Collison nebulizer at an operating pressure of 20 psig. The polydisperse droplets were then dried in a diffusion drier to form a polydisperse aerosol of solid ammonium fluorescein particles. This aerosol was then passed through a single stage impactor to reduce the large particle concentration. The pressure drop across the impactor stage was monitored to verify the magnitude and stability of the aerosol flowrate. Monodisperse particles of the desired size were then produced by electrical classification within the classifier. Lastly, the monodisperse aerosol was diluted to the necessary sampling flowrate using clean, dry dilution air and sampled by the impactor.

The air exiting the impactor was passed through a filter holder housing a 47 mm diameter glass fiber filter which was used to collect any airborne particles not collected by the impactor. Flowrate through the sampling system was provided by a 90 lpm capacity vacuum pump fitted with a primary valve and a recirculation air valve. The pressure drop across a calibrated orifice was used to adjust and monitor the sampling flowrate. A calibrated dry gas meter measured the total volume of the gas sampled during each sampling run.

The collection efficiency of the impactor was defined as that mass fraction of particles exiting the impaction jet which was collected in the liquid

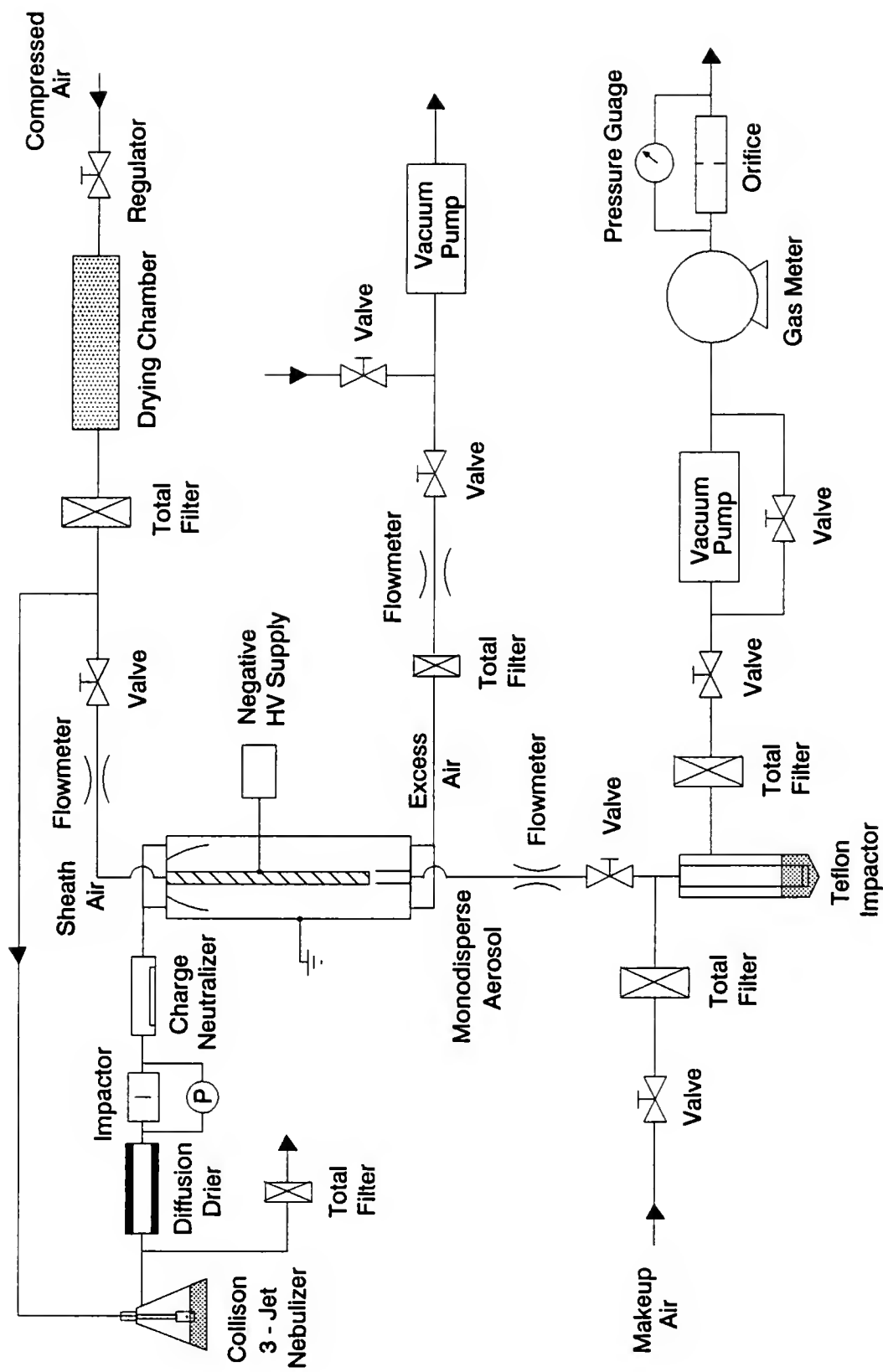


Figure 3-5. Schematic of generation system used for production of calibration aerosols less than 0.5 micrometers diameter.

solution. The total mass exiting the jet was estimated by summing the mass measured in the solution with that mass measured on the 47 mm after-filter.

Collected aerosol mass deposits were quantified fluorometrically using a Turner Model 112 fluorometer calibrated for fluorescein concentrations ranging from one part per billion to 14,000 parts per billion. For the collection efficiency tests, the impactor's liquid solution consisted of 50 ml of 0.1N NH_4OH . At the conclusion of the sampling run, this solution was analyzed directly. To quantify the mass of aerosol collected on the after-filter, the filter was placed in a 100 ml polypropylene container with 50 ml of 0.1N NH_4OH and the container placed in an ultrasonic bath for approximately 20 minutes. Previous analytical tests have shown that virtually 100% extraction efficiency can be achieved using this technique.

Run times for the calibration tests varied depending upon the amount of aerosol mass collected. For the large particle calibration tests, only 10 minute run times were necessary. For the smallest particles generated, however, three hour run times were necessary to achieve sufficient mass deposits for reliable quantitation.

The performance of the impactor was measured as a function of both particle size and sampling flowrate. Thirteen discrete particle sizes were used ranging from 0.05 micrometers to 20 micrometers aerodynamic diameter. For each particle size generated, collection efficiency tests were performed at flowrates of 10, 20, and 30 lpm. Each test series combination of particle size

and flowrate consisted of three separate collection efficiency tests performed under identical test conditions.

Results of the collection efficiency tests are plotted in Figure 3-6.

Individual test results are tabulated in Appendix B. As expected, the impactor's collection efficiency varies as a function of both aerodynamic particle size and sampling flowrate. The right hand side of the collection efficiency curves is of particular importance. Independent of flowrate, the shape of the curves resemble that of conventional inertial impactors with impaction efficiency increasing monotonically with increasing particle size. Clearly, inertial impaction is the predominant collection mechanism for particles in this size range. In accordance with impaction theory, the collection efficiency also increased with increasing flowrate. Measured cutpoints in this region at 10, 20, and 30 lpm were 0.90, 0.53, and 0.46 micrometers aerodynamic diameter, respectively. These measured values compare somewhat favorably with those predicted by standard impactor theory (0.92, 0.63, and 0.42, micrometers).

The overall shape of the complete curves is quite similar to that predicted by the simplified bubbling theory. Impaction is clearly dominant for large particles while molecular diffusion accounts for deposition of small submicron particles. Note also that an absorption minimum occurs in the range of 0.2 to 0.36 micrometers aerodynamic diameter which is approximately the size region predicted by bubbling theory. The position and magnitude of the absorption minimum was also noted to be a function of sampling flowrate. Below

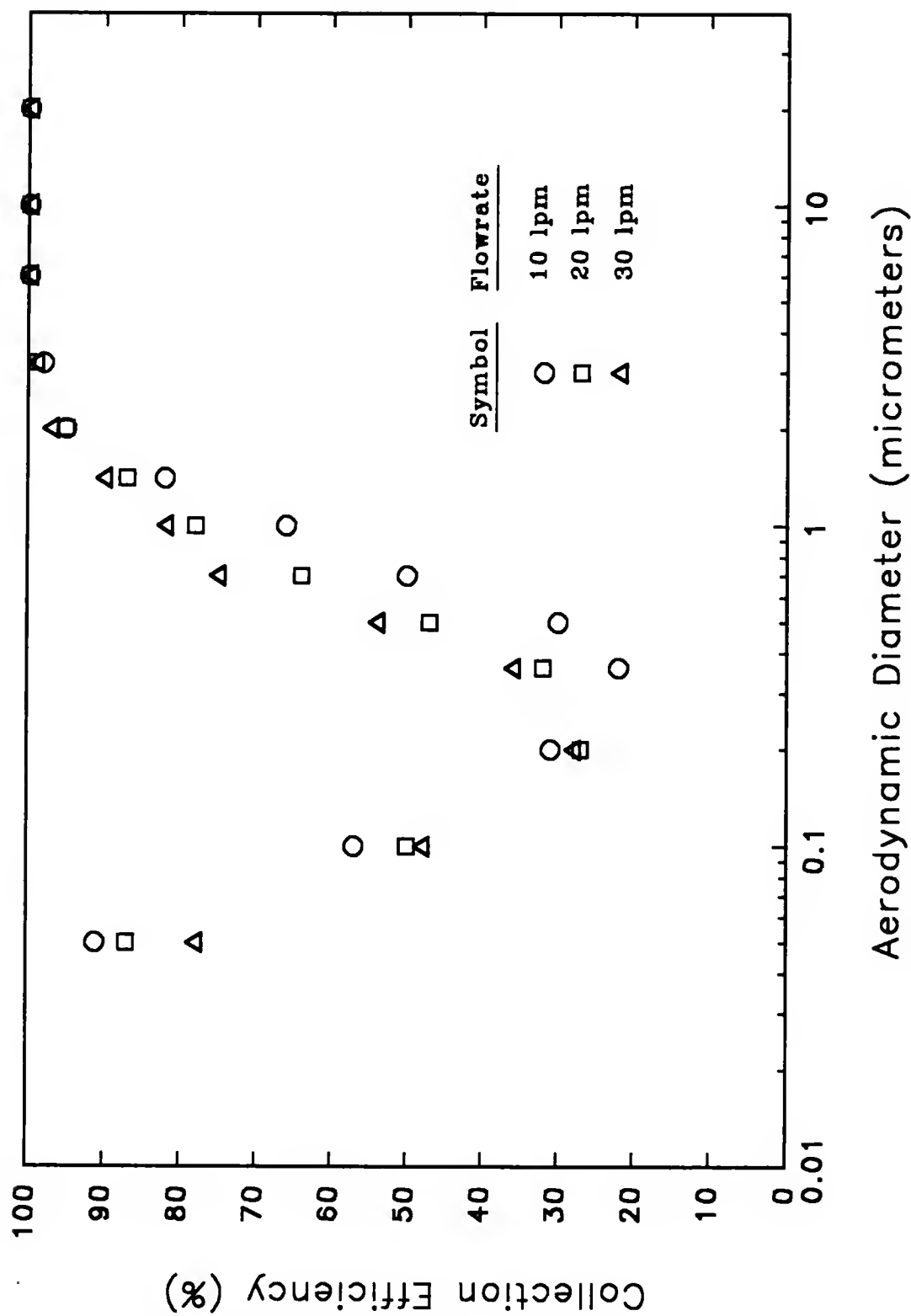


Figure 3-6. Measured particle size collection efficiency curves of single stage impactor.

0.2 micrometers diameter, molecular diffusion dominates the particle absorption and efficiency actually decreases with increasing flowrate. This effect is believed to result as a consequence of the gas' lower residence time within the liquid column at higher flowrates.

In terms of quantitative chemical analysis, it is the overall mass efficiency of the collection device on the aerosol which is of importance. Recall from the measured size distributions in Figure 3-1, that 95% to 99% of the lead-based mass is associated with particles greater than one micrometer aerodynamic diameter. Since the impactor's collection efficiency on these particles is high, integration of the collection efficiency curve over the measured size distribution predicts that the impactor should collect this aerosol with over 95% mass efficiency.

Particle losses through the impactor's inlet section were also quantified during the calibration tests. Following each test series, the impactor was disassembled and the inlet section carefully rinsed with a known volume of 0.1N NH_4OH . Particle mass deposits were quantified by fluorometric analysis of the collected rinse solution. In general, test results showed that inlet losses represent only a small fraction of the sampled aerosol mass. Submicron aerosol deposits were normally below the limit of quantitation of the fluorometric technique. Large particle losses were found to be more significant with losses of 2.2%, 3.2%, and 4.0% measured for particles of 6, 10, and 20 micrometers

aerodynamic diameter, respectively. Subsequent evaluation of the impactor during its actual field use supported these test results.

Based on the calibration tests, it was concluded that the impactor's design met the criteria outlined for the overall instrument design. For the aerosols of interest in the battery industry, the impactor is capable of capturing the aerosols with high collection efficiency. Use of a liquid solution as the impaction substrate eliminates particle bounce and substrate overloading which can exist for solid impaction substrates. Turbulence within the fluid during collection provides for effective dissolution of the lead-based particles as well as providing for a well mixed, homogeneous solution. Unlike solid impaction substrates, the liquid is easily replaced after each sampling test and is compatible with the chosen lead analysis technique. Lastly, the impactor can be easily constructed, is physically compatible with the corrosive nitric acid, and requires little periodic maintenance.

CHAPTER 4 DESIGN OF AIR SAMPLING SYSTEM

Introduction

Accurate quantitation of lead-in-air aerosols requires representative collection and efficient transport of the lead-based particles to the instrument's lead analysis section. Since it is infeasible to capture and analyze an entire aerosol, a fraction of the aerosol must be extracted which reflects the aerosol's particle size distribution, concentration, and chemical composition. Because sampling flowrates may vary from one test to another, the designed instrument's sampling flowrate must be controllable and accurately measured.

Description of Air Sampling System

Figure 4-1 is a schematic of the air sampling components associated with Module 1. A description of these components is given in Table 4-1. In addition to the components shown, Module 1 contains the system's nitric acid reservoir, liquid pumps, and lead analysis system. Referring to Figure 4-1, the vertical orientation of the impactor is compatible with its liquid contents and minimizes particle transport losses from the Module 1 inlet to the impaction stage. The Module 1 sampling system consists of a set of sharp-edged

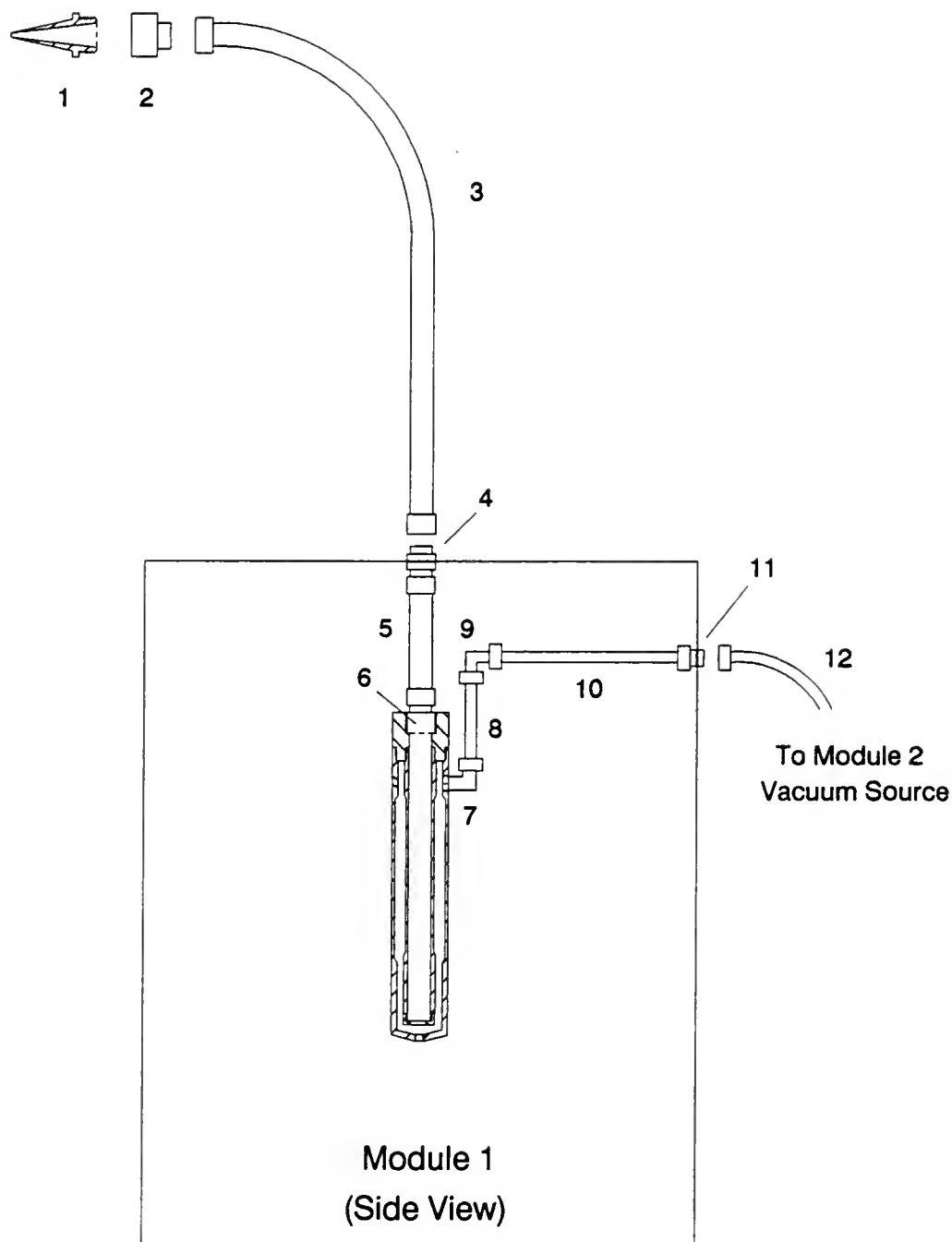


Figure 4-1. Schematic of Module 1 air flow system. Component descriptions are provided in Table 4-1.

Table 4-1
Description of Module 1 Air Flow Components

<u>Figure 4-1 Code</u>	<u>Description</u>
1	3/16, 1/4, or 3/8 in. Diameter SS Nozzle
2	SS Female Connector, 1 in. Tube - 1 in. NPT(F)
3	SS Flexible Hose, Teflon Lined Cajon, Inc. Model SS-16BHT-36
4	SS Bulkhead Union, 1 in. Tube - 1 in. Tube
5	SS 3/8 in. O.D. Tube, 4.25 in.
6	SS Male Connector, 1 in. Tube - 1 in. NPT(M)
7	SS Male Elbow, 3/8 in. Tube - 1/2 NPT(M)
8	SS 3/8 in. O.D. Tube, 3 7/8 in.
9	SS Union Elbow, 3/8 in. Tube
10	SS 3/8 in. O.D. Tube, 7 7/8 in.
11	SS Bulkhead Union, 3/8 in. Tube
12	3/8 in. Polypropylene Tubing, 6 ft.

nozzles, sampling probe, and impactor in addition to their associated tubing and compression fittings.

Sampling of indoor air (non-ducted airstreams) is used to evaluate occupational lead exposures. For indoor air measurements, the sampling nozzles and sampling probe are not needed. Instead, a sharp-edged nozzle has been fabricated from a one inch diameter stainless steel tube and attaches to the top of Module 1. Its vertical orientation allows for direct collection and transfer of airborne particles to the collector with minimal transport losses.

Unlike indoor air sampling, the sampling of ducted airstreams such as recirculation airstreams requires sampling from a horizontal duct with high associated air velocities. As will be discussed, accurate particle sampling from a flowing airstream requires that a thin-walled nozzle be oriented directly into the airstream. In addition, the sampling flowrate must be controlled such that the nozzle inlet velocity equals that of the freestream velocity at the point of sampling. Depending upon the measured freestream velocity, a nozzle of 3/16, 1/4, or 3/8 inch diameter can be used.

Sampling from horizontal ducts requires that the sampled aerosol must undergo a 90 degree change of direction prior to entering the instrument's vertical collection section. The sampling system contains a 39 inch long flexible probe which is used for this purpose. The probe consists of a teflon inner lining surrounding by a stainless steel braid. The teflon lining is compatible with elevated temperatures up to 400 °F and is resistant to corrosion. The probe's

0.88 inch inner diameter matches that of the impactor inlet tube thus minimizing particle transition losses due to air expansion or contraction.

Air flow through Module 1 is provided by connection to the vacuum source located in Module 2. As shown in Figure 4-2, Module 2 contains an air filter, mass flow controller, and cooling fan. Component descriptions are presented in Table 4-2. The cartridge filter contains both activated charcoal and fibrous filters and serves to protect the mass flow controller and sampling pump from any particles remaining in the airstream. A high capacity vacuum pump with teflon coated wetted surfaces provides the driving force for air flow through the sampling system. As shown in Figure 4-2, this pump has been fitted with a recirculation valve to reduce the load to the pump.

The sampling flowrate in the lead-in-air monitor is controlled using a surplus Tylan Model FC-262 mass flow controller which has been adapted for use in the system. The FC-262 is a 0-50 slpm capacity unit with flowrate controlled by an internal solenoid valve. Selection of the flowrate is normally provided by manual adjustment of a potentiometer to supply a 0 to 5 VDC command signal to the flow controller. Feedback circuitry within the flow controller continually adjusts the valve setting so that the actual air flow equals the desired flowrate selected by the user. A measure of the actual flowrate is provided by a corresponding 0 to 5 VDC output signal which is normally read by the user from a digital panel meter. For adaptation to the prototype monitor, the potentiometer input circuit and output display were removed and the

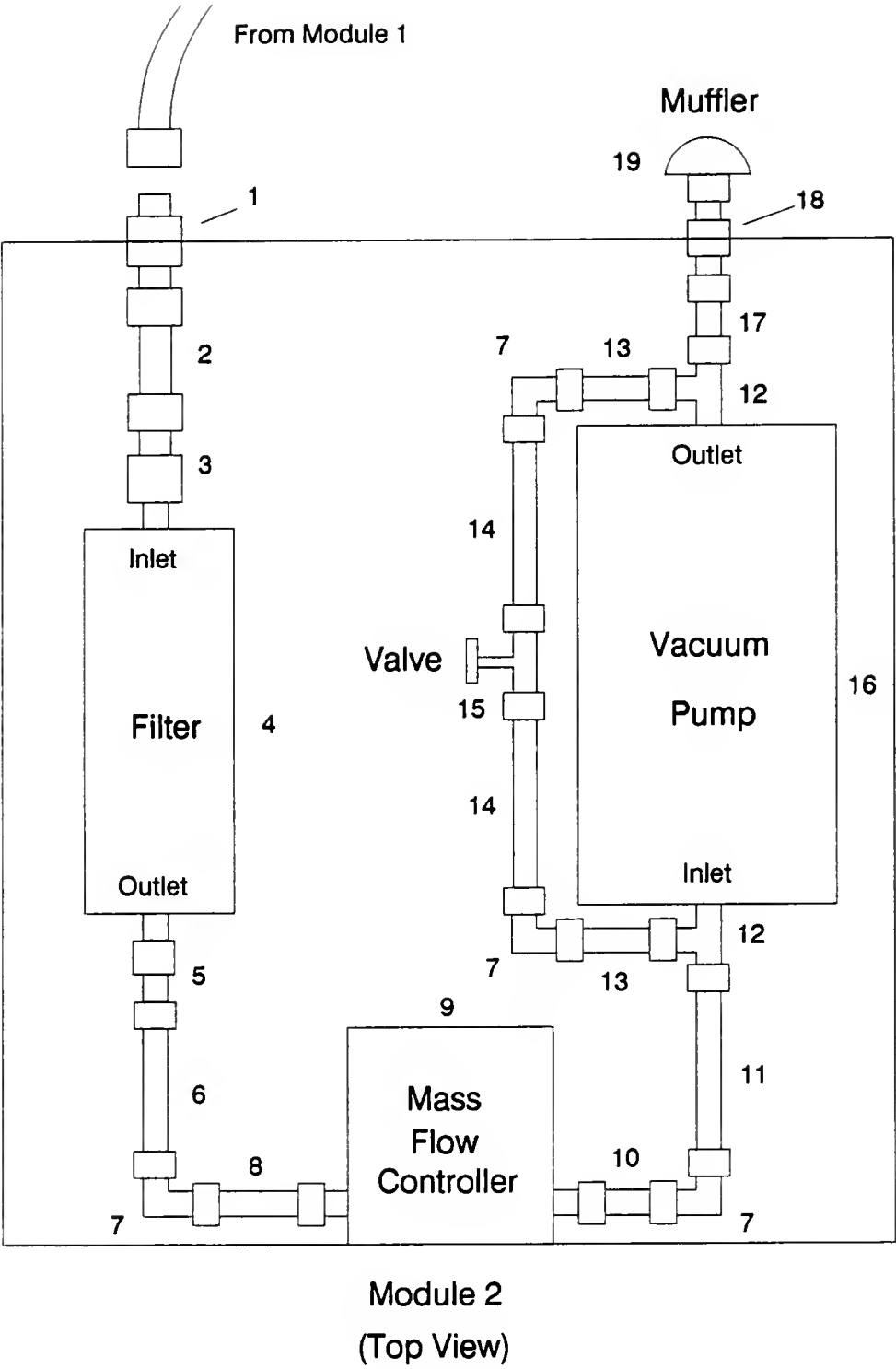


Figure 4-2. Schematic of Module 2 air flow system. Component descriptions are provided in Table 4-2.

Table 4-2
Description of Module 2 Air Flow Components

<u>Figure 4-2 Code</u>	<u>Description</u>
1	SS Bulkhead Union, 1/2 in. Tube - 1/2 in. Tube
2	SS 1/2 in. Tube, 2 3/16 in.
3	SS Female Connector, 1/2 Tube - 3/8 NPT(F)
4	Disposable Air Filter Koby Inc., Model "Junior King"
5	SS Female Connector, 3/8 in. Tube - 3/8 NPT(F)
6	3/8 in. Polypropylene Tube, 6 5/16 in.
7	SS Elbow, 3/8 in. Tube - 3/8 in. Tube
8	SS 3/8 in. Tube, 1 3/4 in.
9	Mass Flow Controller, 0-50 slpm Tylan Corp., Model FC262
10	SS 3/8 in. Tube, 2 in.
11	3/8 in. Polypropylene Tube, 5 3/8 in.
12	SS Male Tee Run, 3/8 in. Tube - 3/8 NPT(M)
13	SS 3/8 in. Tube, 2 5/8 in.

Table 4-2--continued

<u>Figure 4-2 Code</u>	<u>Description</u>
14	SS 3/8 in. Tube, 2 9/16 in.
15	Valve, Swagelok Corp., Model B1RS6
16	Vacuum/Pressure Pump, 1/4 hp, teflon lined 3.2 cfm, 4A at 0 psi Thomas Industries, Model 727CM39-TFE
17	3/8 in. Polypropylene Tube, 2 1/4 in.
18	SS Bulkhead Female Connector, 3/8 in. Tube to 3/8 in. NPT(F)
19	Muffler, Koby Inc., Model M-1-N

command signal is now supplied by an analog output channel from the data acquisition and control system. The flow controller's output signal is read by a corresponding analog input channel on the data acquisition and control system.

Figure 4-3 is a complete wiring diagram of the Module 2 circuitry.

Component descriptions, functions, and commercial sources are listed in Table 4-3. Table 4-4 provides connector pin descriptions. A standard 120 VAC, 60 hz source supplies power to the mass flow controller power supply, vacuum pump, and cooling fan. Power to the flow controller is supplied by a 15 VDC, 200 mA power supply. The power supply is protected using a 0.5 amp fuse. The sampling pump is internally protected against current overload and thus requires no additional fusing. On/off control of the air pump and the cooling fan is provided by use of a solid state relay capable of handling load currents up to 25 amps. The relay itself is activated through application of a 3-32 VDC signal which is compatible with standard TTL digital command signals. All Module 2 input and output signals are communicated by a standard 9 pin ribbon cable to the Kiethley 575 data acquisition and control system.

Prior to its use, the Tylan mass flow controller had been repaired and recalibrated by the manufacturer. Its condition and calibration were then verified in the laboratory. At a variety of flowrates and upstream and downstream pressures, the actual mass flowrate was measured as a function of selected mass flowrate. For these tests, a National Bureau of Standards traceable Gillian Model D-80028 bubble flow meter was used as a flow

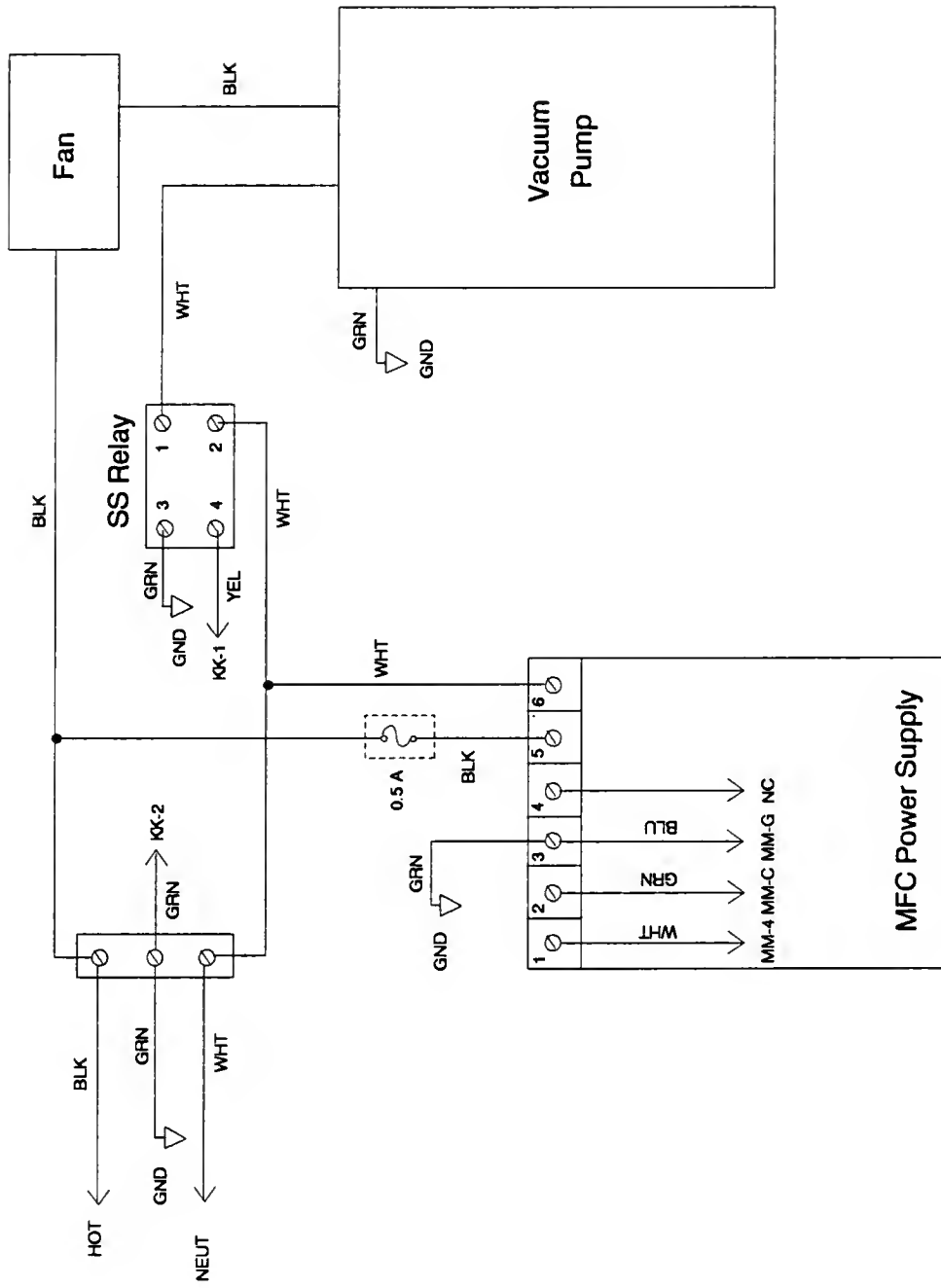


Figure 4-3. Wiring diagram of Module 2 electrical components. Component descriptions are provided in Table 4-3. Connector pin functions are presented in Table 4-4.

Table 4-3
Module 2 Electrical Component Listing and Description

<u>Component</u>	<u>Description</u>
Barrier Terminal Block	3 Contact, Double Row, 20A Cinch, Inc. type 3-141
Solid State Relay	SSR Series, 240 VAC/25A 3-32 VDC input Potter and Brumfield Type SSR-240D25
Appliance Plug	250/115 VAC, 10/15A Feller, Inc. type 6100-31
Cooling Fan	115 VAC, 0.15A 34 cfm at 0 in H ₂ O S.P. Comair/Rotron type SV2A1
Fan Filter Screen(2)	Anodized, for 3.5 in fan Fan-S/Qualtek type 06325-B
Vacuum/Pressure Pump	115 VAC, 1/4 hp, teflon lined 3.2 cfm, 4A at 0 psi Thomas Industries type 727CM39-TFE
Power Supply	Dual Voltage (-15/+ 15 VDC), 200 mA Sola, Inc. type 85-15-2120
Fuse	1 1/4 X 1/4 in, 0.5A slow-blow SPC Technology type F-500SS-0.5
Fuse Block	Screw termination SPC Technology type FB1PS
Connector KK	9 pin D-Sub, crimp style plug SPC Technology type DEC-9P
Connector MM	22 position, card-edge connector

Table 4-4
Module 2 Electrical Connector Pin Descriptions

<u>Pin#</u>	<u>Color</u>	<u>575 Channel</u>	<u>Connection</u>	<u>Function</u>
KK-1	YEL	DIGOUT8	Relay	Pump, fan on/off
KK-2	GRN	Ground	Ground	Ground
KK-3	BRN	ANOUT1	LL-A	MFC Command Signal 0-5 VDC
KK-4	RED	ANIN1	LL-3	MFC Output Signal "High", 0-5 VDC
KK-5	BLK	ANIN0	LL-2	MFC Output Signal "Low", 0-5 VDC
MM-2	BLK	ANIN0	KK-5	MFC Output Signal "Low", 0-5 VDC
MM-3	RED	ANIN0	KK-4	MFC Output Signal "High", 0-5 VDC
MM-4	WHT	---	Power	+ 15 VDC Supply
MM-A	BRN	ANOUT1	KK-3	MFC Command Signal 0-5 VDC
MM-C	GRN	---	Power	Common
MM-G	BLU	---	Power	-15 VDC Supply

calibration standard. For each operating pressure and flowrate range, six separate measurements were made. As shown in Figure 4-4, the correlation between the actual mass flowrate and the desired flowrate was generally good. In the 10-30 slpm flowrate range of interest, the agreement was within 2%. Note that conversion from actual to standard flowrate must account for the fluid's temperature and pressure at the point of sampling. Conversion from standard temperature as defined by the mass flow controller's manufacturer (0 °C) to standard temperature as defined by occupational regulations (25 °C) must also be taken into account. In the prototype lead-in-air monitor, these conversions take place automatically through the instrument's software.

Prediction of Particle Sampling and Transport Efficiency

Accurate measurement of lead-in-air concentrations requires that lead-based particles be sampled, transported, collected, and analyzed efficiently. Design of an efficient particle sampling and transport system depends primarily on the particle size distribution expected, the fluid properties at the point of sampling (such as temperature and velocity), and the air sampling flowrate required by the instrument's particle collection section. Efficient design, calibration, and field testing of a separate sampling system was beyond the scope of this project. As a preliminary approach, a particle sampling and transport system was adapted from EPA Reference Method 12, "Determination of Inorganic Lead Emissions from Stationary Sources." In the forthcoming

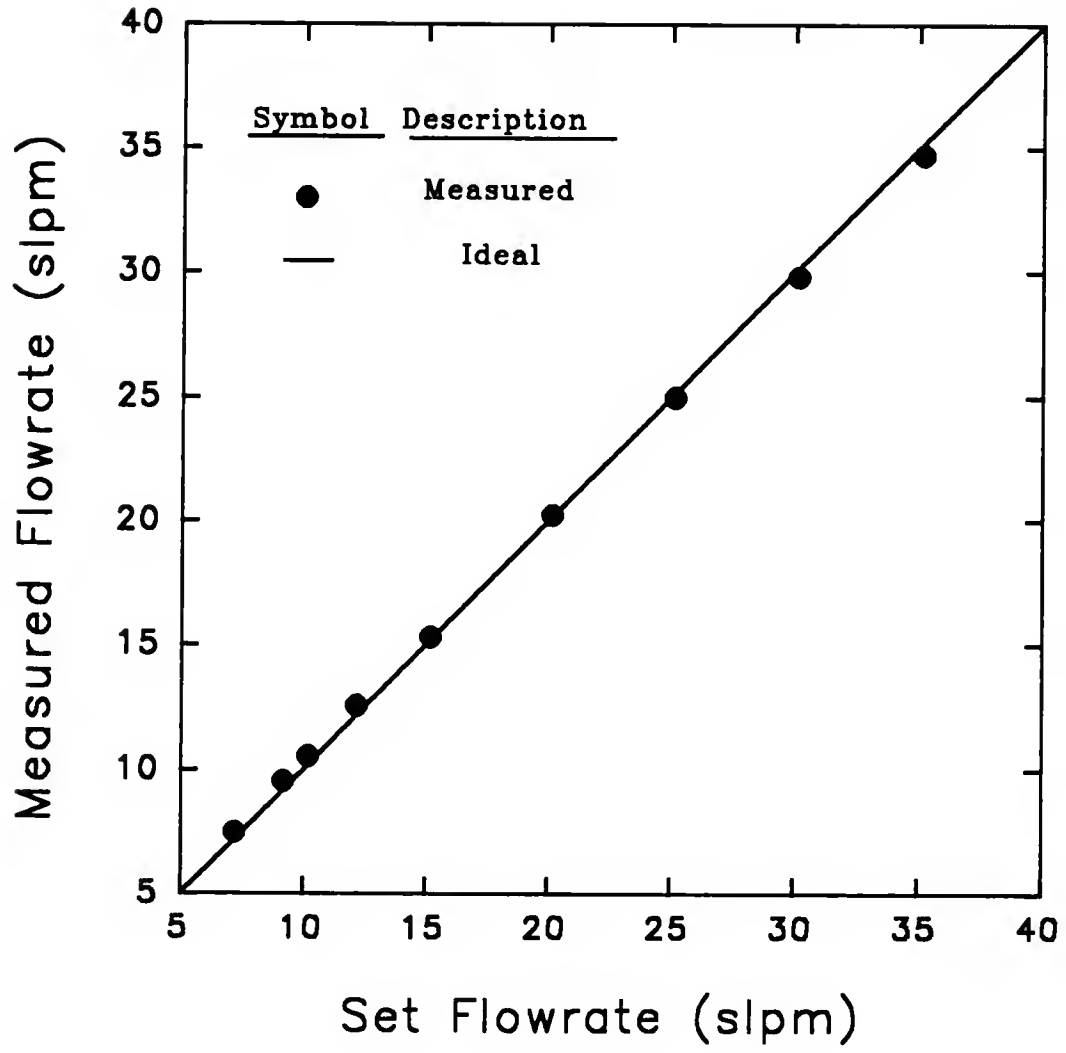


Figure 4-4. Measured performance of Module 2 mass flow controller.

sections, the sampling and transport efficiencies of this design will be predicted based on published theoretical and empirical studies. Alternatives to this sampling and transport system will be discussed.

Sampling from Still Air

Evaluation of occupational exposures to lead-in-air concentrations represents sampling from an airstream at zero or low velocity. Because particles possess higher stopping distances at higher velocities, sampling and transport losses during still air sampling can be negligible when compared to sampling from ducted, high velocity airstreams. There exist, however, two types of still air sampling biases which can result particularly with regard to the large particle distributions characteristic of the battery industry.

Efficient still air sampling requires use of a vertically oriented sampling tube. When the inlet air velocity is low, a sampling bias can result due to high settling velocities characteristic of particles with large aerodynamic diameters. At low sampling velocities, this results in an overestimation of particle concentrations. An additional bias can result due to the effect of particle inertia on particle trajectory. For sampling in still air, curved streamlines result as air is accelerated towards the inlet nozzle. Due to their inertia, particles will be unable to follow their respective streamlines and will be displaced a distance which may be large with respect to the dimensions of the inlet. Inertial effects of this type can result in an underestimation of actual particle mass concentrations.

Based on these possible biases, various criteria have been developed for the design of air samplers. Davies (1968) developed inlet criteria based on parameters of nozzle inlet diameter, sampling flowrate, and particle aerodynamic diameter. Agarwal and Liu (1980) predicted less than 10% sampling losses if the probe diameter (D_t) meets the following criterion

$$D_t > 20 \tau^2 g \quad (4-1)$$

where τ is the particle relaxation time and g is the gravitational constant.

Equation 4-1 was used to predict inlet losses of the 0.88 inch nozzle used for still air sampling of the lead-in-air monitor. Results showed that particles up to 60 micrometers aerodynamic diameter would be collected without significant error. Particles greater than 60 micrometers would be collected with less than 90% efficiency. The equal diameter of the nozzle the inlet section ensures that particle transport losses should be minimized.

Sampling from Ducted Air Streams

Unlike sampling from still air, sampling from ducted airstreams can result in significant sampling biases particularly with regard to the high velocity airstreams and large particle distributions characteristic of the battery industry. Moreover, the horizontal arrangement of ventilation ducts requires that a captured aerosol must be transported a short distance to the instrument which also involves a change in the aerosol's flow direction. As a result, particle

losses within the aerosol transport system may significantly reduce the mass concentration detected by the lead analysis section. The factors affecting particle sampling and transport biases will be discussed.

The purpose of the aerosol sampling system is to obtain a representative aerosol sample from the airstream of interest. In ducted airstreams, this involves use of a sampling nozzle and probe to extract a representative aerosol sample and transport it to the instrument's collection section. Sampling bias can occur, however, due to particles' inertia in flowing airstreams. Similar to the sampling from still air, sampling criteria have been developed for efficient sampling from ducted airstreams. These criteria are generally of three types. First, sharp-edges inlet nozzles must be used to obtain representative air samples. Blunt-edged orifices can disrupt the airflow entering the nozzle resulting in nonrepresentative particle collection and high inlet losses (Vincent and Gibson (1981), Vincent et al. (1982), Vincent et al. (1985), Stevens (1986), Vincent (1987), Vincent and Gibson (1988)). The second criterion is that the nozzle must be oriented directly into the oncoming airstream. Changes in the sampled air direction due to nozzle misalignment are a function of particle inertia, the misalignment angle, and the probe diameter (Durham and Lundgren, 1980). Lastly, the sampling should be performed under isokinetic conditions (nozzle inlet velocity equal to freestream velocity). Anisokinetic sampling can result in significant large particle sampling biases whose magnitude and direction depend on the velocity ratio and the particle Stokes number. Note

that isokinetic sampling requires prior measurement of the airstream's velocity at each of the desired sampling points. When sampling, achieving desired nozzle inlet velocities requires proper combination of nozzle diameter and sampling flowrates.

Achieving representative aerosol sampling does not ensure efficient transport of the aerosol from the inlet nozzle to the particle collector. Particle losses can occur both in the inlet nozzle as well as in the transport section. Deposition can take place due to molecular diffusion, thermophoretic effects, electrostatic effects, gravitational effects, and inertial effects. For the sampling of aerosols of interest in the battery industry, only gravitational and inertial mechanisms will result in significant transport losses.

Numerous theoretical and experimental studies have been conducted in relation to understanding particle transport in sampling tubes. In general, these studies agree on the recognized parameters of importance (such as particle Stokes number, sampler geometry, and fluid conditions) but often vary widely in terms of their conclusions and recommendations. Theoretical predictions of transport losses requires extensive mathematical modeling of both fluid flow fields and particle trajectories within the flow fields. The validity of these models is often not confirmed. Differences in sampler geometries and flowrates can result in significantly different predicted losses. Experimental loss measurements are generally more practical but differences in sampler

geometries, operating flowrates, and particle properties make comparison of separate studies difficult.

In the lead-in-air monitor's sampling system, particle transport losses during duct sampling can occur either in the inlet nozzle or in the flexible sampling probe. To predict nozzle inlet losses, a brief review of pertinent literature was made. Although the actual loss will depend on nozzle diameter, sampling flowrate, and particle size, the literature suggests that large particle losses may exceed 5% by mass. In the lead-in-air monitor, additional nozzle losses might also be expected due to the airflow's slight expansion from the nozzle outlet diameter of 0.75 inches to the 0.88 inch diameter of the sampling probe. The magnitude of the losses in the expansion section will depend on sampling flowrate and particle size.

Losses in the sampling probe can more accurately be predicted than those associated with the inlet nozzles. Considering the flexible probe oriented as in Figure 4-1, the probe can be divided into three main sections for analysis: an 8 inch long horizontal section, a 23 inch long section with a 14.5 inch radius of curvature, and a 8 inch long vertical section. The particle losses in these sections were considered separately. These calculations were simplified by the fact that laminar flow conditions exist in the 0.88 inch diameter probe even at a flow of 30 lpm. Turbulent flow conditions alter the nature of the fluid flow field and particle trajectories making accurate loss predictions more complex.

Particle deposition in the 8 inch long horizontal section occurs primarily due to the force of gravity. During their transit time within the tube, particles may settle a distance sufficient to be removed from the airstream. The magnitude of these losses was predicted based on a study by Thomas (1958) on sedimentation losses in horizontal cylindrical tubes. For laminar flow conditions, the transport efficiency (penetration) can be predicted

$$P = 1 - \frac{2}{\pi} \left[2 \phi \sqrt{1 - \phi^{2/3}} + \arcsin \phi^{1/3} - \phi^{1/3} \sqrt{1 - \phi^{2/3}} \right] \quad (4-2)$$

$$\phi = \frac{3 \pi L V_s R}{8 Q}$$

where P is the fractional penetration, L is the tube length, V_s is the particle settling velocity, R is the tube radius, and Q is the volumetric flowrate through the tube.

This relationship was evaluated for the 8 inch long section as a function of particle size and sampling flowrate and is presented graphically in Figure 4-5. Independent of flowrate, particles less than 10 micrometers aerodynamic diameter can be transported through this section with fairly high efficiency. Particle penetration above 10 micrometers, however, is predicted to decrease fairly rapidly as a function of particle size. Note also that the lower sampling flowrate results in lower transport efficiency due to the gas' longer residence time within the horizontal tube. Efficient transport through this section,

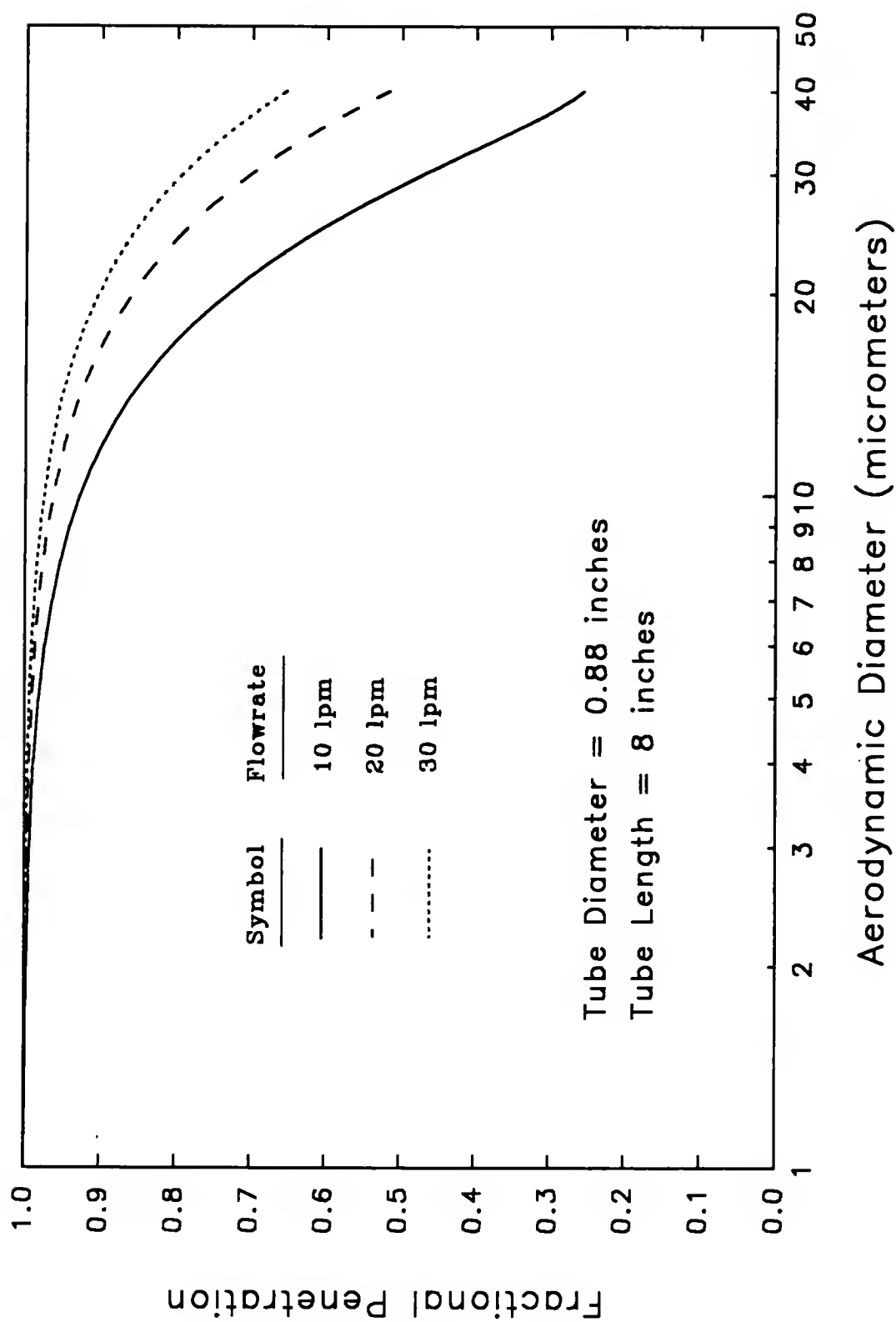


Figure 4-5. Theoretical particle transport efficiency through the sampling probe's horizontal section.

therefore, would imply the use of high sampling flowrates and tubes of smaller diameter.

Unlike losses in horizontal sampling tubes, particle losses in 90° bends are due primarily to inertia and centrifugal forces on the airborne particles. Due to their inertia, particles will be unable to follow their respective streamlines directly and may be displaced a sufficient distance to impact on the duct walls. Air flow around the bend can also establish a rotational flow pattern normal to the mean direction of flow. This rotational profile can result in additional particle losses around the bend. Cheng and Wang (1981) developed a relationship for transport efficiency in a 90° bend as a function of particle Stokes number

$$P = 1 - STK \quad (4-3)$$

$$= 1 - \frac{\rho_p D_p^2 Q}{18 \mu \pi R^3}$$

This relationship holds for laminar flow conditions and is plotted in Figure 4-6 as a function of particle size and flowrate. Similar to losses in the horizontal section, losses around the bend are minimal for particles less than 10 micrometers aerodynamic diameter. Particles greater than 20 micrometers aerodynamic diameter are difficult to transport efficiently. Note also the effect of sampling flowrate on transport efficiency. Higher flowrates correspond to larger

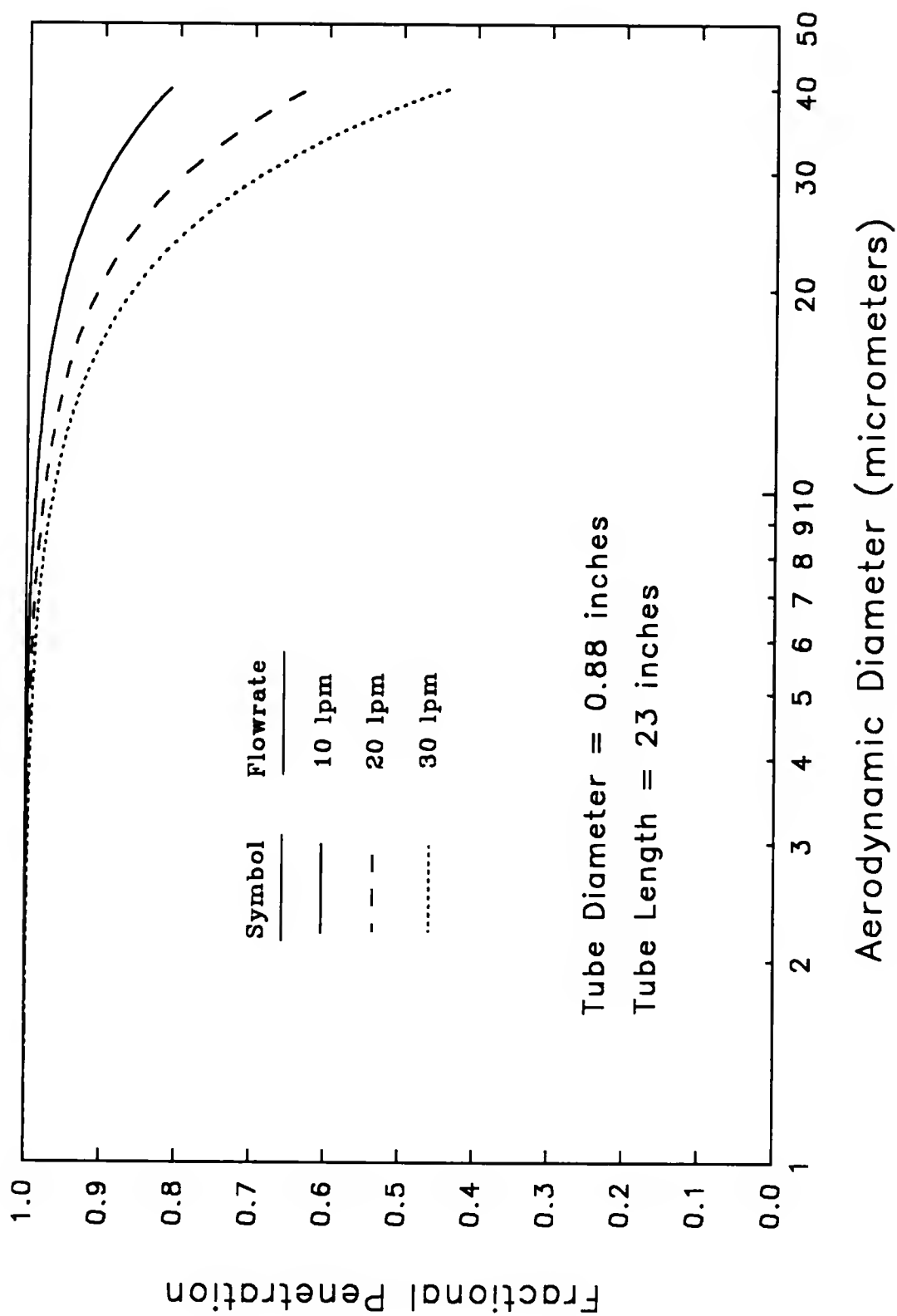


Figure 4-6. Theoretical particle transport efficiency through the probe's 90° bend.

stopping distances which would result in greater particle deposition. Inspection of equation 4-3 would dictate use of low sampling flowrates and large diameter tubes.

Note that the effects of flowrate and tube diameter on bend losses are contradictory to those required for efficient horizontal transport. For a given tube diameter, higher flowrates result in higher horizontal transport efficiency but lower transport efficiency around the bend. Conversely, lower flowrates minimize bend losses but maximize losses in the probe's horizontal section. Combined losses within these two sections are thus relatively independent of sampling flowrate. The magnitude of these losses depends primarily on aerodynamic particle size.

Particle losses within the probe's final 8 inch long vertical section are predicted to be minimal. Since the tube is oriented vertically, no horizontal deposition is possible. Operation of the probe in the laminar flow regime also eliminates the possibility of turbulent deposition which can be significant for particles with high Stokes numbers in turbulent flow fields. This prediction of low particle losses in this section is supported by the low impactor inlet losses measured during the impactor's laboratory calibration.

It can be concluded, therefore, that use of the proposed sampling nozzle and probe may not allow efficient transport of large particles to the lead-in-air monitor's collection and analysis section. Combined losses within the nozzle, nozzle outlet, and sampling probe may be prohibitively high. It should be

noted, however, that these loss calculations may significantly overestimate particle losses. Theoretical loss calculations normally assume that when a particle contacts a surface, it is removed from the airstream. Use of liquid aerosols during experimental loss tests would support these calculations. In reality, solid particles contacting solid surfaces at high velocity may experience only fractional sticking efficiencies. Reentrainment of deposited particles also results in higher particle transport efficiency.

It is apparent that special sampling considerations may need to be given to the on-line measurement of ducted airstreams in the battery industry. Conventional stack sampling techniques and equipment may not enable representative sampling and transport of these large aerosols. As possible alternatives, three sampling approaches could be investigated. First, the ductwork at the monitoring point of interest could be modified to include a short vertical section so that the entire flow is directed downward. A sharp-edged sampling nozzle attached vertically to the lead-in-air monitor could then isokinetically extract a representative sample with minimal inlet and transport losses. Alternatively, a small flexible test section attached to a high volume vacuum source could isokinetically extract a portion of the major duct flow and direct this secondary airstream downward over the monitor's sampling nozzle. Aerosol not sampled by the monitor could then be returned to the major airstream or filtered prior to its exhaust to the atmosphere. This concept is similar is design by Baron (1988) and Ball and Mitchell (1990) for efficient

transport of coarse aerosols to particle sizing instrumentation. As a final alternative, the conventional sampling nozzle could be replaced with a precollector designed to remove all particles greater than 10 micrometers aerodynamic diameter. The remaining fraction could then be transported efficiently to the monitor's collection section. In the human respiratory tract, particles less than 10 micrometers represent the size fraction capable of penetrating to the alveolar region and thus represent the best measure of the actual lead exposure hazard. Particles larger than 10 micrometers are deposited in the upper regions of the respiratory tract and are absorbed in the bloodstream with much lower efficiency. Recent developments in 10 micrometer size selective inlets indicate that this design approach is feasible. Use of a precollector to evaluate worker exposure, however, must first meet with approval by the Occupational Health and Safety Administration.

CHAPTER 5 DESIGN OF LEAD ANALYSIS SECTION

Introduction

As discussed, accurate evaluation of lead-in-air concentrations requires efficient aerosol sampling, transport, collection, and accurate analysis of the collected aerosol's lead content. Previous sections have outlined the techniques required to ensure representative aerosol sampling, transport, and collection. This chapter will outline design criteria for the lead analysis section, briefly review available analytical techniques, and describe in detail the analytical technique chosen for incorporation into the lead-in-air monitor.

Design Criteria

Selection of the instrument's lead analysis technique was based on careful consideration of several design criteria. First, the technique must be specific for elemental lead and capable of accurately quantifying lead concentrations ranging from 50 ppb to 9000 ppb. It was desired that the technique's response be fairly linear over a wide range of concentrations and the technique display adequate repeatability. Because the collected aerosol sample's composition is not entirely lead, the analytical technique should

possess minimal physical and chemical interference from these other components. The technique must also be physically compatible with the chosen particle collection substrate. It was important that the selected analytical technique require little periodic maintenance or recalibration. Finally, the selected technique must meet cost requirements and be adaptable to convenient automation.

Based on these criteria, a thorough review of pertinent literature was made with regard to selection of the instrument's analytical technique. There exist a wide variety of analytical techniques available for the quantitation of lead either in solid or liquid phase. These techniques include both conventional and graphite furnace atomic absorption spectroscopy, ion chromatography, inductively coupled argon plasma spectroscopy, x-ray and electron techniques, colorimetry, neutron activation analysis, and electrochemical techniques. For an automated device, most of these techniques were eliminated from consideration either due to their high cost, level of complexity, inadequate lead detection capabilities, or failure to adapt themselves to convenient automation. For example, conventional atomic absorption is incapable of detecting sub-ppm levels of lead in solution. Although graphite furnace atomic absorption possesses the necessary detection capabilities, its use would have greatly increased the complexity and cost requirements of the monitor. In addition, the response of both conventional and graphite furnace atomic absorption displays a fairly limited range of linearity. Other techniques such as colorimetry involve a

number of chemical handling steps which would have been inconvenient to automate.

Ultimately, an electrochemical method of lead analysis was regarded as most appropriate for the lead analysis. Because metals display high electroactivity, their presence in solution can be detected over a wide range of concentrations. Electrochemical techniques display high linearity and repeatability. In particular, the electrochemical technique of polarography was selected as that which best met the outlined design criteria. In forthcoming sections, the principle of polarography will be reviewed and its application to lead-in-air monitor described.

Description of Polarographic Analysis

Electrochemical analysis is based on the observed behavior of electroactive species in solution when subjected to an electric potential gradient. For the detection of a particular species of interest (such as metals), a varying potential is applied to the solution through a conductive electrode. If at a particular potential a species is oxidized or reduced, an electric current will flow at the electrode. Since the rate of electron transfer is a function of the number of redox reactions occurring at a given potential, the measured current produced is proportional to the concentration of that component in solution. Because electroactive species display characteristic redox potentials, the observed potential at which the redox reaction occurs can be used to identify

the component of interest. By applying a range of potentials to a sample and noting the potential and resulting current of several redox reactions, simultaneous quantitation of several solution components is possible. This analytical technique is commonly referred to as voltammetry.

Although the study of voltammetry is not new, recent advances have led to the development of reliable, commercially available instruments suitable for trace metal detection. Current designs now incorporate mercury drops as the active working electrode surface. Since a fresh mercury drop is produced for each separate sample analysis, carry over contamination of the working electrode is virtually eliminated. Recent advances in microprocessor technology have also led to increasing measurement sensitivity and instrument capabilities.

Commercially available analyzers allow the user to select from various applied potential waveforms which optimize detection of the component of interest. For many metals (including lead) part per billion detection capability is routine.

Based on a review of available electrochemical detectors, an EG&G Model 303A static mercury drop electrode was purchased along with an accompanying Model 264A control unit. The Model 303A contains a mercury-based working electrode, a silver/silver chloride reference electrode, and a platinum counter electrode which are all immersed in a static cell containing the solution of interest. The Model 303A also contains a 5 lb capacity mercury reservoir and a delivery system for dispensing individual mercury drops of repeatable size.

Control of the Model 303A electrode system is provided through communication with the Model 264A control unit. The Model 264A sets the Model 303A operating parameters, provides for automatic sequencing of the analysis steps, receives and conditions the resulting output signals, and communicates the current versus voltage scan results to an x-y recorder. The user is able to select from a variety of scan types including normal direct current, normal pulse, and differential pulse polarography as well as direct current and differential pulse stripping analysis. Although these techniques possess varying degrees of capabilities and applications, differential pulse stripping analysis provides for the most sensitive detection of lead in solution. This mode, therefore, is used exclusively for lead quantitation in the lead-in-air monitor.

Quantitation of lead in solution by stripping analysis typically involves the following sequence of steps. First, high purity nitrogen is bubbled through the liquid sample at a flowrate of approximately 40 cc per minute for approximately 3 minutes. This deaeration step removes the electroactive interference of any dissolved oxygen in the solution. The Model 303A then dispenses a single mercury drop of selectable size which hangs on the tip of a glass capillary immersed in the solution. A constant electric potential is applied to the mercury drop whose magnitude is more negative than the half-wave potential of the analyte of interest. This results in an electro-reduction of the analyte onto the surface of the mercury drop. To ensure repeatable mass transfer rates during

this step, the solution is stirred and deposition is performed for a fixed time period (such as 3 minutes). The preconcentration step is responsible for the technique's low detection limit for many electroactive species.

At the conclusion of the deposition step, the stirrer is turned off and the electroactive species is stripped from the electrode by making the electrode more positive and oxidizing the species back into solution. In this stripping step, varying the potential at a constant rate (such as 10 mV/sec) allow different species to oxidize at different times. In differential pulse stripping analysis, the current measured during each species' oxidation reaches a maximum value that is proportional to the concentration of each species that was deposited.

As an example, Figure 5-1 presents the results of a typical analysis of 40 ppm lead, cadmium, and copper standards in 0.1N nitric acid. The position of the peaks (with potentials referenced to the Ag/AgCl electrode) identifies the species present in solution and the peak height for each species is proportional to its concentration in solution.

To ensure reliable test results of trace metal analysis, special experimental techniques were observed during all polarographic analyses. To minimize sample contamination and species absorption losses, all polarographic cells were made of teflon and were acid washed with 6M nitric acid prior to use. Metal standards were prepared from 1000 ppm stock metal standards certified by the manufacturer to within 1% accuracy. Serial dilutions were performed

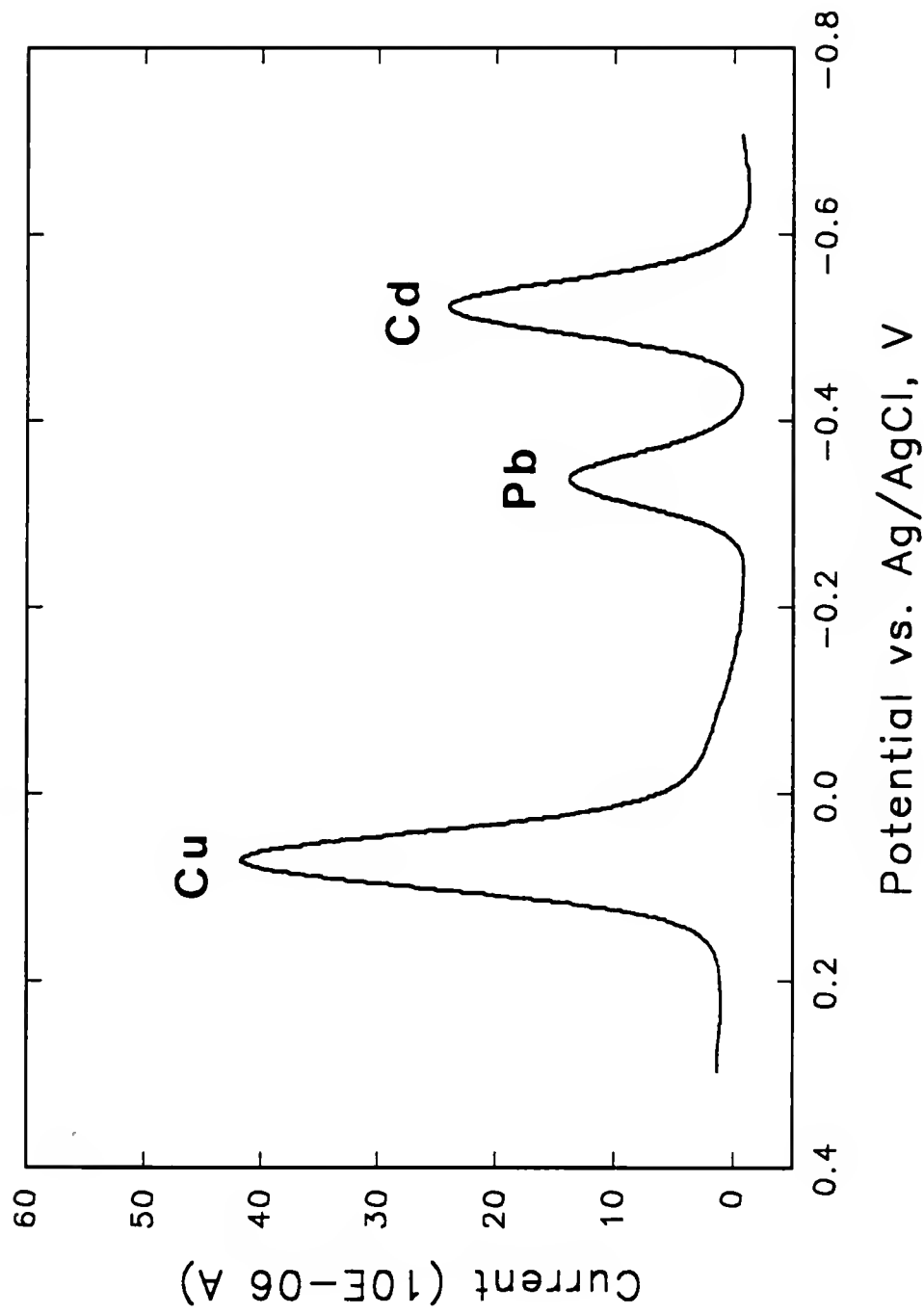


Figure 5-1. Polarographic response to 40 ppm concentrations of lead, cadmium, and copper in 0.1N HNO₃.

using positive displacement pipettes with disposable tips rated to within 2% accuracy. Dilute nitric acid solutions were prepared using trace metal grade concentrated nitric acid diluted with distilled, deionized water with a measured resistivity of 17×10^6 ohm-cm. Only certified, triple distilled mercury and ultra-high purity nitrogen were used in the polarograph.

If polarographic stripping analyses are performed under carefully controlled conditions, repeatable test results can be obtained over a wide range of concentrations. Figure 5-2 presents the instrument's response as a function of lead concentration in 0.1N nitric acid. Each data point represents the average of 6 separate tests performed under identical test conditions. The technique's precision (as expressed by the relative standard deviation) was measured to be approximately 2%.

Considerable attention was devoted to identification and subsequent optimization of the parameters which affect the polarographic's response to trace lead concentrations in solution. Factors affecting the deposition step were identified to be initial deposition potential, mercury drop size, deposition time, and solution stirring dynamics. The instrument's response during the stripping phase was found to vary with the potential's rate of change (scan rate) and the height of the differential wave applied to achieve oxidation. The overall response was also found to vary with the condition of the Ag/AgCL reference electrode, solution temperature, and the concentration of the nitric acid solution used.

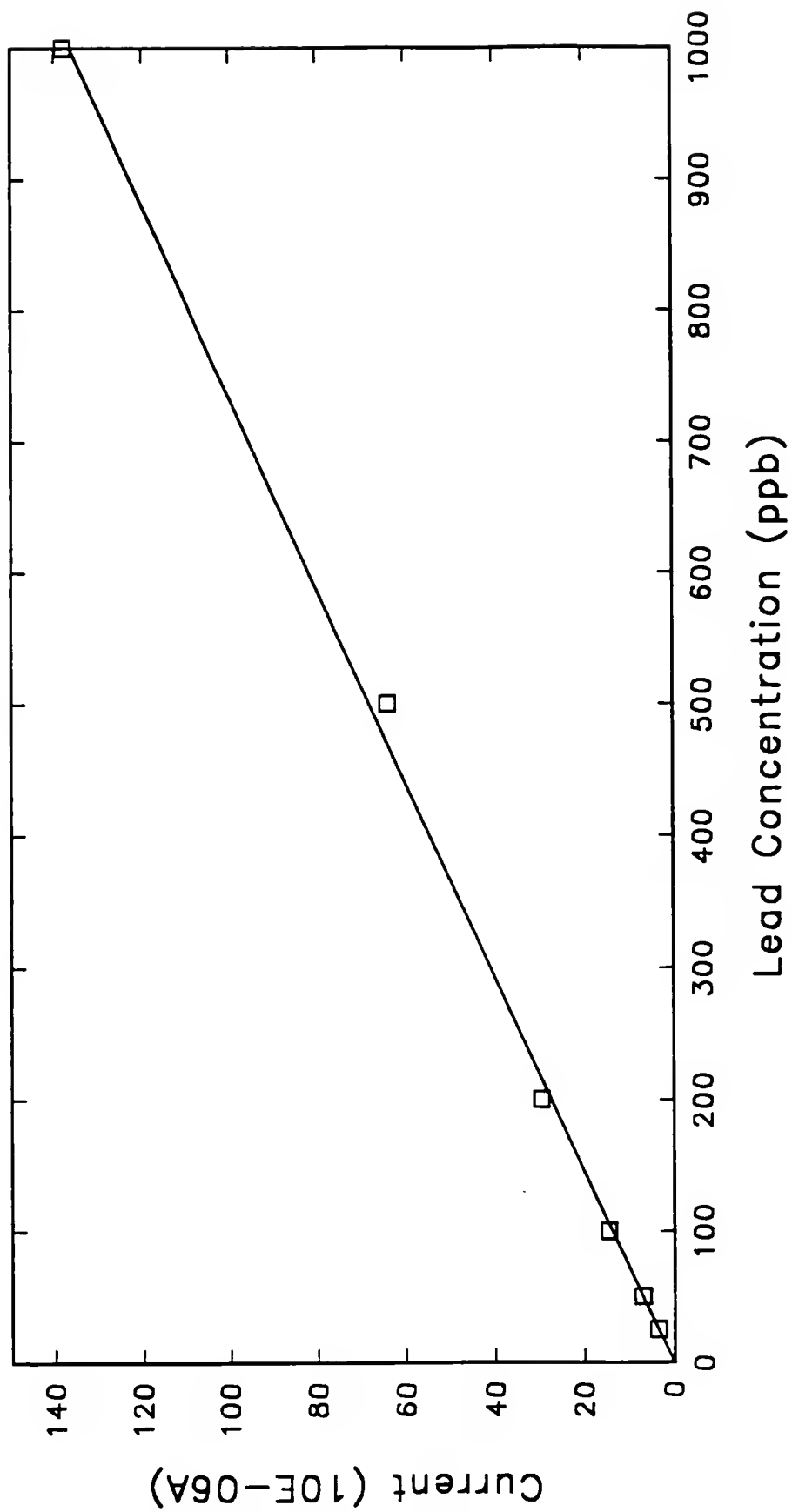


Figure 5-2. Typical polarographic response as a function of lead concentration.

Within limits, these variables can be controlled by proper selection of the polarograph's operating parameters. However, complete control of all of these variables in an automated monitor is impractical to achieve. In stripping analysis, the instrument's response to a given lead standard was noted to vary as much as 50% during successive test series. Ultimately, this variability was virtually eliminated by including an internal standard (200 ppb copper) in the nitric acid electrolyte.

Use of the internal copper standard was developed based on numerous observations of the polarograph's response to several species in solution. During the analysis of equal mass concentrations of lead, copper, and cadmium, it was noted that the ratios of the resulting peak heights were essentially constant regardless of the operating parameters of the instrument. For example, although reducing the deposition time during stripping analysis reduced the magnitude of each element's peak, the respective peak ratios remained essentially constant. Similar behavior was noted by changing the mercury drop size, nitric acid concentration, analysis scan rate, solution temperature, and solution stirring dynamics. Thus, if the instrument response to a known concentration of an analyte is measured, the concentration of a different species can be calculated from its observed peak height and the known peak ratio. During the course of the project, the instrument's response to equal lead and copper concentrations was measured as a function of concentration and instrument's operating parameters. For 156 separate

measurements, the ratio of the copper to lead peak height was measured to average 2.33 with a relative standard deviation of 1.8%. Assuming the use of a 200 ppb copper internal standard, the concentration of lead in solution can thus be calculated using the following relationship

$$\text{Lead Conc. (ppb)} = \frac{\text{Lead Peak Height}}{\text{Copper Peak Height}} \times 2.33 \times 200 \text{ ppb} \quad (5-1)$$

Use of an internal standard in the lead-in-air monitor offers several advantages. First, the need for periodic calibration of the analytical section is eliminated since the instrument essentially calibrates itself during each sample analysis. Analytical accuracy is no longer sensitive to variables which would be difficult to control within required limits. Lastly, use of the internal copper standard provides an internal quality control check during each analysis. If the instrument's response to the copper standard is outside the expected range, it serves as an indication that either the electrolyte was prepared incorrectly or that a possible instrument malfunction may exist.

To determine the value of this design approach, lead standards of concentrations ranging from 0 to 8000 ppb were prepared in 0.5M nitric acid with a 200 ppb copper internal standard. The instrument's response to these solutions was then measured and Equation 5-1 used to predict the actual lead concentration. For each concentration, six separate measurements were performed under identical test conditions. Figure 5-3 presents the results of the 0 to 200 ppb lead measurements in comparison to the instrument's ideal

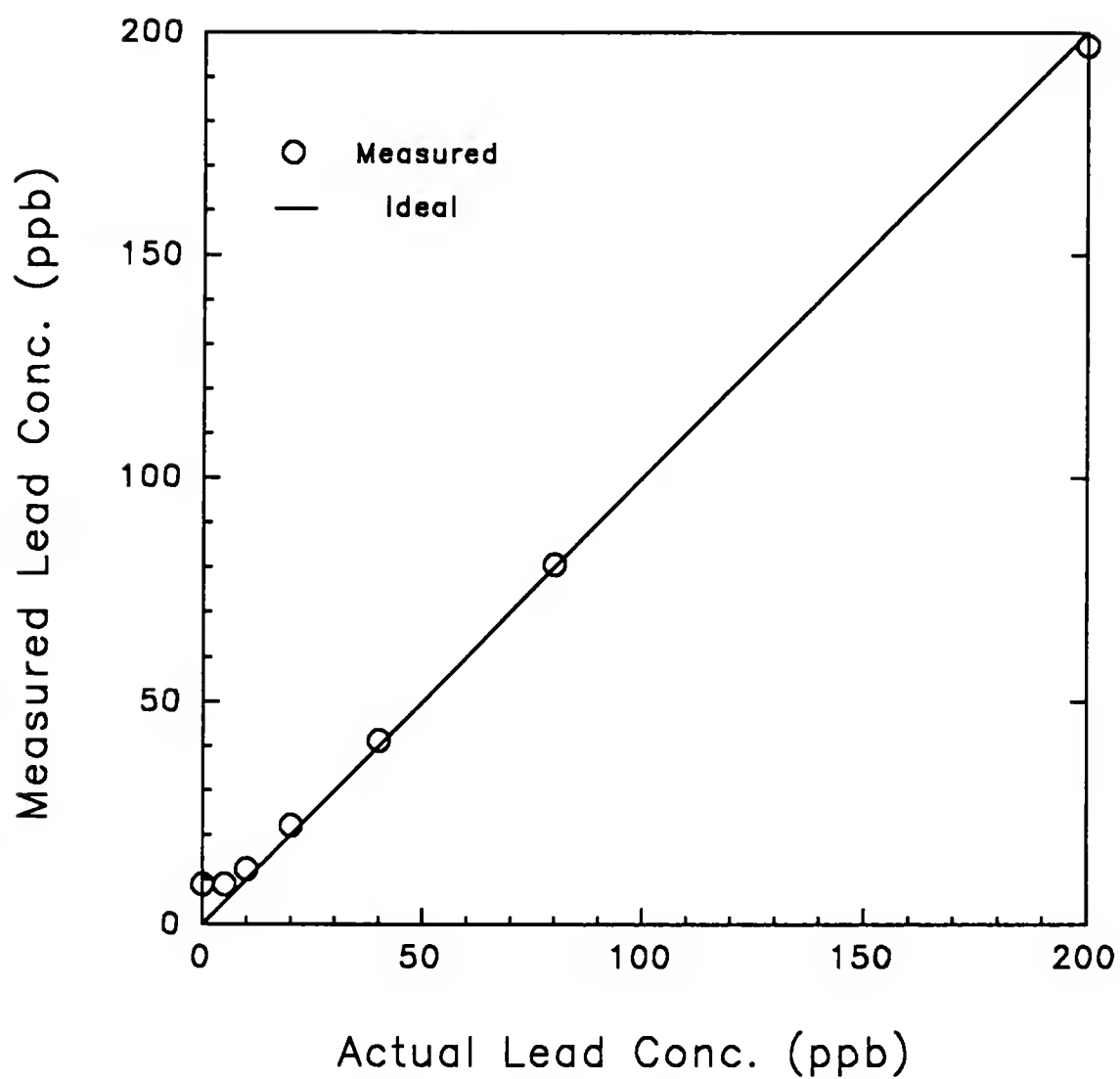


Figure 5-3. Polarograph response to 0-200 ppb lead concentration using 200 ppb internal copper standard.

response. It can be seen that agreement between the measured and ideal lead concentration is quite good for lead concentrations greater than or equal to approximately 20 ppb. The observed response at 0 ppb is due to the presence of hydronium ions in solution which provide a residual current overlapping the position of the lead peak. At a 0 ppb lead concentration this interference typically provides a calculated lead concentration of 5 to 15 ppb. For lead concentration above 20 ppb, however, the contribution of the hydronium ions is insignificant. A minimum quantitation limit of 20 ppb lead is thus reported for the monitor's analytical section.

Figure 5-4 presents the results of the 0 to 8000 ppb lead response tests. Note that use of the internal copper standard is accurate even over this wide range of concentrations. A summary of these test results is tabulated in Table 5-1 including the technique's precision as expressed by the relative standard deviation. For lead concentrations ranging from 20 ppb to 8000 ppb, the precision was calculated to be approximately 2%.

Analysis of Field Bulk Samples

In any quantitative chemical analysis, it is important to ensure that the analytical technique chosen be compatible with the sample to be analyzed. Physical and chemical interferences associated with the sample's measurement should be noted and effort made to minimize them in order to achieve reliable test results. In the case of the lead-in-air monitor, it was important to ensure

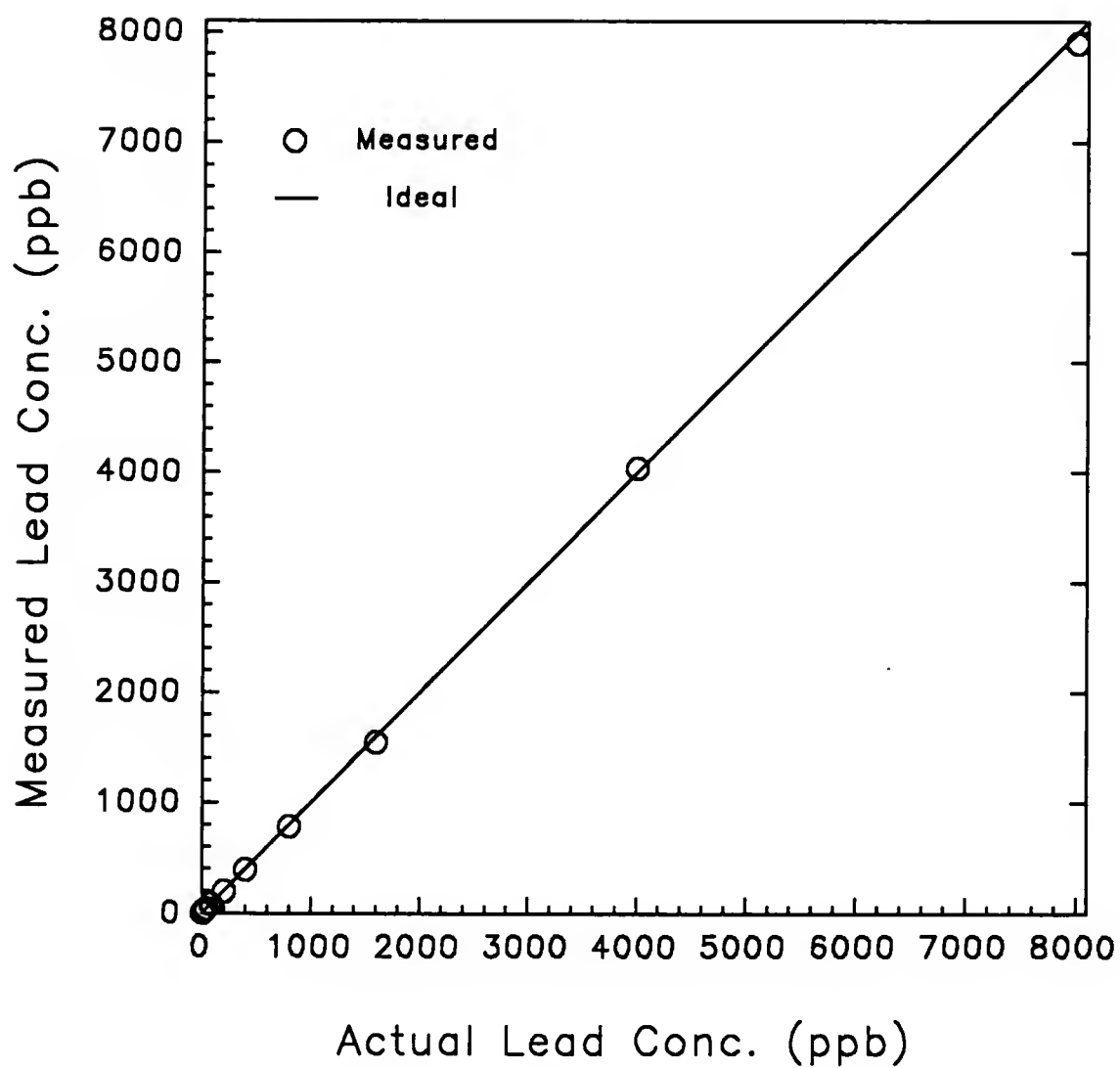


Figure 5-4. Polarograph response to 0-8000 ppb lead concentration using 200 ppb internal copper standard.

Table 5-1
Polarograph Response Using Copper Internal Standard

<u>Actual Lead Conc. (ppb)</u>	<u>Polarograph Response Lead Conc. (ppb)</u>	<u>RSD(%)</u>
0	9	8.3
5	9	7.0
10	12	5.9
20	22	3.1
40	41	0.7
80	81	0.4
200	197	0.8
400	389	0.7
800	783	0.8
1600	1546	1.8
4000	4037	2.1
8000	7900	2.4

that the expected aerosol's composition be compatible with polarographic analysis.

During battery manufacturing, airborne particles are produced primarily by mechanical breakup (dispersion) of lead oxide powder, lead oxide paste, and the lead-based plate material. It can thus be expected that the chemical composition of the aerosol produced from these materials is virtually identical to that of its parent material. Bulk samples of lead oxide powder, paste, and plate material were obtained at the battery plant and returned to the laboratory for analysis in order to identify any potential analytical interferences.

During these tests, a known mass of each material was dissolved in nitric acid solution and analyzed using the polarograph. For comparison purposes, these samples were also analyzed using a graphite furnace atomic absorption (GFAA) spectrophotometer manufactured by Perkin-Elmer (Model 5100). The GFAA is more widely used for trace metal analysis than the polarograph and thus represents an accepted criterion upon which to base the polarograph's accuracy. For each test series, the GFAA was calibrated for lead concentrations in its linear operating range of 0 to 40 ppb by autodilution of a 100 ppb lead stock standard solution. For comparison purposes, the diluent consisted of 0.5M nitric acid with a 200 ppb copper internal standard. To minimize matrix effects associated with lead measurement by GFAA analysis, a fixed quantity of a matrix modifier ($0.2 \text{ mg NH}_4\text{H}_2\text{OPO}_4 + 0.01 \text{ mg Mg(NO}_3)_2$) was injected with each $5 \mu\text{l}$ sample.

The results of these comparison tests are presented in Table 5-2.

Analysis was first performed on certified laboratory samples of lead nitrate, lead monoxide, and lead acetate. Based on the known formula weights of these materials, each sample was prepared to obtain an expected lead concentration of 100 ppb. As shown in Table 5-2, both the polarograph and the GFAA measured these solutions with reasonable accuracy. Based on the three replicate analyses for each material, the polarograph displayed somewhat better repeatability than did the GFAA.

Similar solutions were prepared from the bulk materials collected at the battery plant. Although the lead content of the lead monoxide powder can be assumed with some confidence, its actual percentage in the lead paste can only be estimated based on discussions with the battery plant personnel. Results shown in Table 5-2 generally verified these estimates. Similar to the analysis of the certified laboratory samples, these results indicated fairly good agreement between the polarograph and the GFAA. More important, no interference was observed during the polarographic analysis of these battery plant samples. It was concluded, therefore, that no interference can be expected during analysis of any airborne particles produced from these bulk materials.

Table 5-2
Comparison Tests Between Polarograph and GFAA

<u>Description</u>	Lead Conc. (ppb) (RSD)		
	<u>Expected</u>	<u>Polarograph</u>	<u>GFAA</u>
<u>Laboratory Samples</u>			
Blank	0	11 (6.2%)	1 (18.2%)
100 ppb Pb Std (certified)	100	102 (0.6%)	104 (7.8%)
108 ppb Pb Std (certified, no Cu)	100	-- --	98 (4.5%)
108 ppb PbO (certified)	100	98 (0.0%)	94 (2.8%)
108 ppb Pb-Acetate (certified)	100	99 (0.6%)	92 (7.1%)
<u>Field Samples</u>			
108 ppb PbO from battery plant	100	101 (0.0%)	94 (3.8%)
108 ppb Pb-paste from battery plant	95-100	95 (0.6%)	98 (1.8%)
108 ppb Plate material from battery plant	90-95	93 (2.1%)	94 (3.7%)

Description of Modifications to the Polarograph

Introduction

In previous sections, the suitability of polarography for the instrument's analytical section has been discussed. The polarograph is commercially available, meets costs requirements, possess the necessary lead detection limits and measurement range, and displays no analytical interferences from the aerosols of interest to the battery industry. To achieve the design goals of the complete lead-in-air monitor, however, several modifications were made to the polarograph to adapt it to automated control. These modifications were made primarily to eliminate the need for manual selection of the polarograph's operating parameters, manual selection of the proper measurement range, and manual retrieval and interpretation of test results.

Run/Stop Control of Model 264A

On the Model 264A control unit, initiation of an analysis run is performed manually using a DPDT momentary contact pushbutton switch (SW1) located on the main 264A circuit board. Similarly, termination of an analysis is performed manually using a separate pushbutton switch (SW2). To provide for remote control of these run/stop functions, a printed circuit board was designed, constructed, and has been installed within the Model 264A. A wiring diagram of this circuit is presented in Figure 5-5 along with component

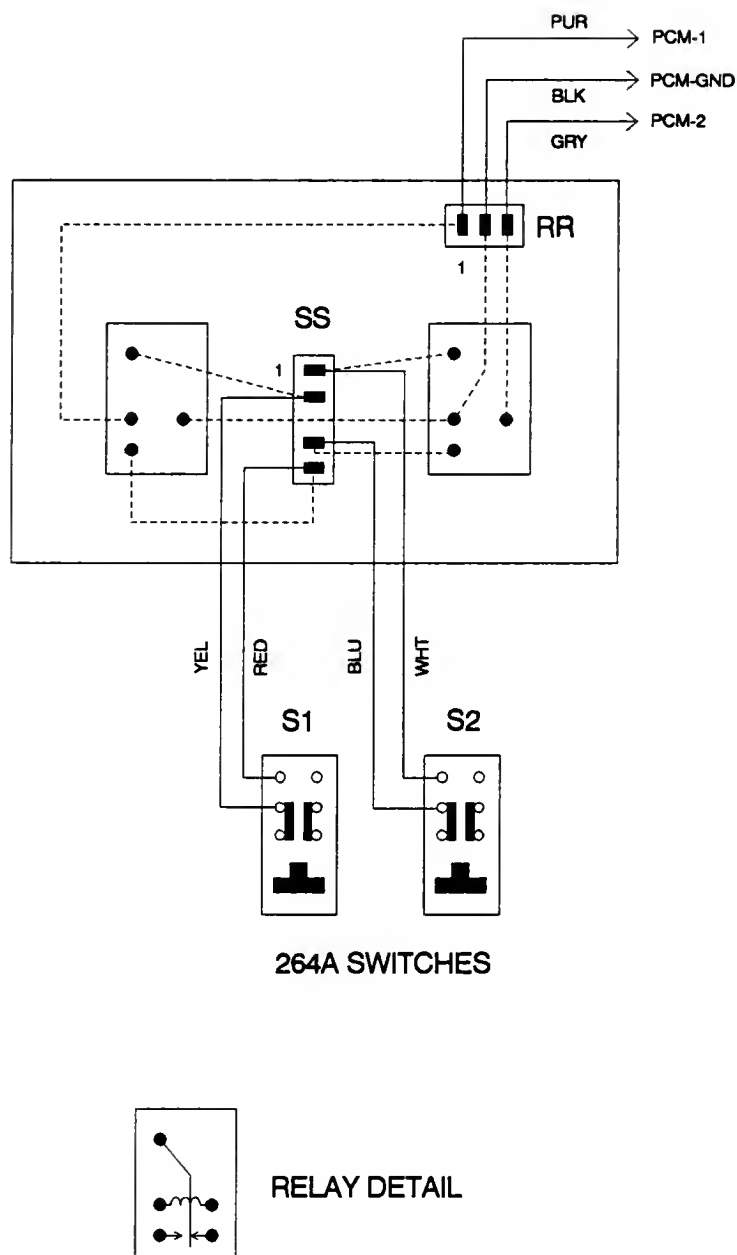


Figure 5-5. Schematic of printed circuit board which provides run/stop functions. Component descriptions are provided in Table 5-3. Connector pin functions are described in Table 5-4.

descriptions in Table 5-3. Connector pin functions are presented in Table 5-4. The "run" circuit uses a SPDT electromechanical relay in parallel with SW1 to provide for initiation of the run function. Activation of the relay's coil is provided by a 5 VDC command signal from a Kiethley power control module (PCM3) which has also been installed within the Model 264A. Software control of the PCM3 channel releases the relay once the polarographic analysis cycle has begun. Similarly, the manually operated stop function has been automated through a separate electromechanical relay activated by a separate PCM3 channel.

Figure 5-6 presents the wiring diagram of the PCM3 control module with accompanying component descriptions and channel functions listed in Tables 5-5 and 5-6, respectively. Although the PCM3 is capable of providing both AC and DC power control for up to 16 separate channels, only 8 channels providing DC control are required in the lead-in-air monitor. Activation of each channel is provided through the data acquisition and control system's software.

Description of Decade Resistance Board

During a polarographic scan, the electrical current produced during a redox reaction is proportional to the concentration of the species undergoing the reaction. In the Model 264A, this measured current undergoes a current-to-voltage conversion using an operational amplifier (U2 on 264A circuit board) with a maximum output voltage of 5 VDC. Since the current can vary over five

Table 5-3
Component Listing and Description of RUN/STOP Board

<u>Component</u>	<u>Description</u>
Electromechanical Relays	5 VDC, 72 mA, SPDT DIP 2A at 125 VDC Radio Shack Type 275-243
Connector RR	5 pin Pout Header Friction Lock Amphenol Type 640456-6
Connector SS	5 pin Pout Header Friction Lock Amphenol Type 640456-6

Table 5-4
RUN/STOP Board Connector Pin Functions

<u>Pin</u>	<u>Color</u>	<u>PCM Channel</u>	<u>Function</u>
RR-1	PUR	PCM-1	Activates RUN Relay
RR-2	BLK	PCM-GND	Ground
RR-3	GRY	PCM-2	Activates STOP Relay
SS-1	WHT	-	STOP Switch (S2) Contact 1
SS-2	YEL	-	RUN Switch (S1) Contact 1
SS-3	BLU	-	STOP Switch (S2) Contact 2
SS-4	RED	-	RUN Switch (S1) Contact 1

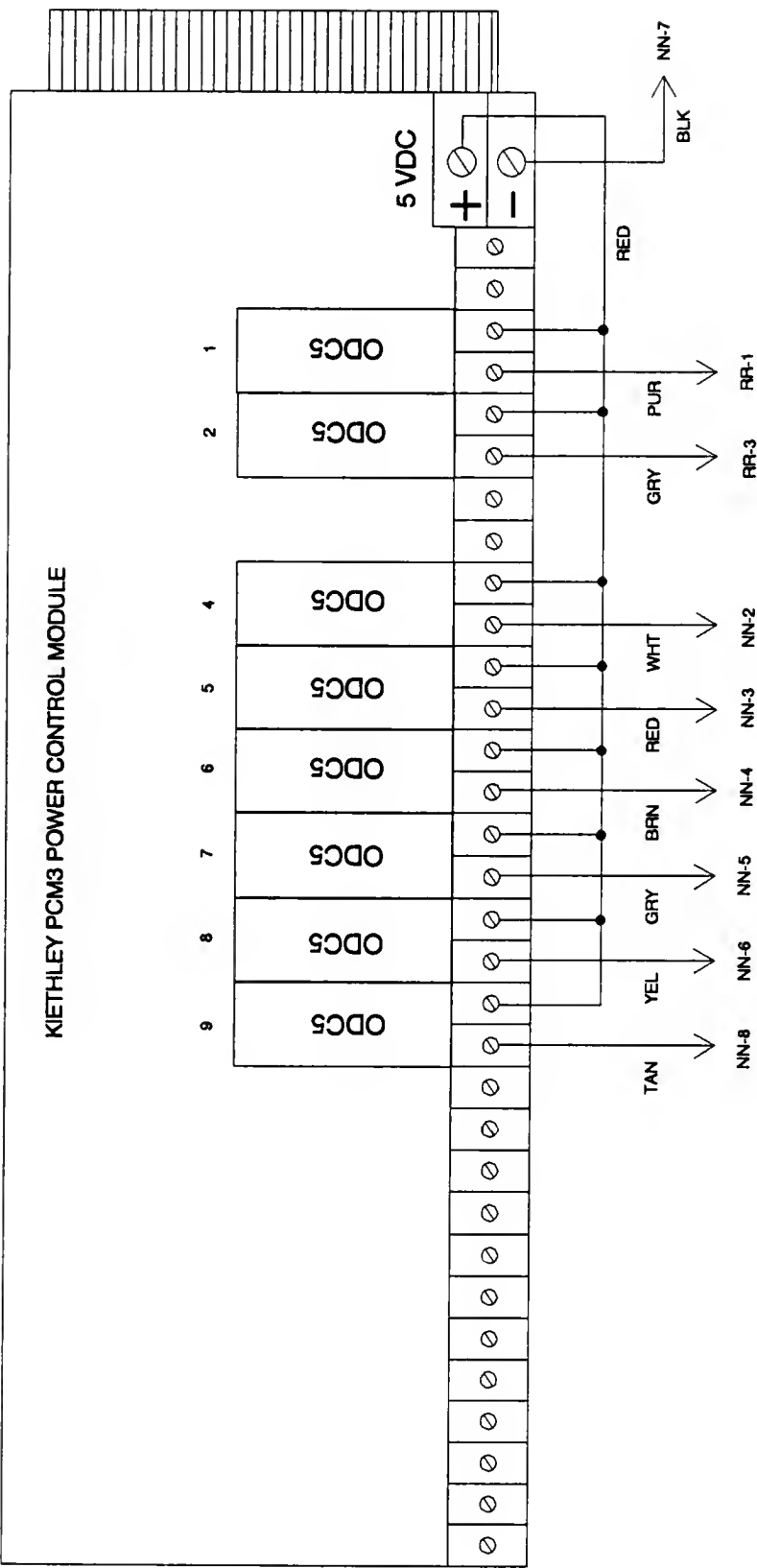


Figure 5-6. Wiring diagram of Kiethley PCM3 power control module. Component descriptions are provided in Table 5-5. Channel functions are listed in Table 5-6.

Table 5-5
Component Listing and Description of PCM3 Board

<u>Component</u>	<u>Description</u>
Solid State Relays	5 VDC Output 3-14 VDC input, 3A at 60 VDC Potter and Brumfield Type SSR-ODC5
Power Control Board	Kiethley PCM3 Module

Table 5-6
Description of Various PCM3 Channels

<u>PCM3 Channel</u>	<u>Color</u>	<u>Connection</u>	<u>Function</u>
1	PUR	RR-1	Activates RUN Relay
2	GRY	RR-3	Activates STOP Relay
3	WHT	MM-2	Activates Decade PCB Relay 5
4	RED	MM-3	Activates Decade PCB Relay 4
5	BRN	MM-4	Activates Decade PCB Relay 3
6	GRY	MM-5	Activates Decade PCB Relay 2
7	YEL	MM-6	Activates Decade PCB Relay 1
8	TAN	MM-8	Activates Decade PCB Relay 6

orders of magnitude, the gain of the amplifier is controlled through a feedback loop providing five decades of resistance. Varying this feedback resistance is equivalent to changing the instrument's measurement range. On the 264A, this gain switch is selected manually based on the maximum concentration expected during each sample analysis. If the actual concentration is higher than expected, however, an over-range condition occurs and no useful results can be obtained from the analysis. Conversely, if the gain switch is set too low, the output voltage produced by the amplifier will be too low to produce reliable test results. In either case, the operator must make the proper adjustment to the gain switch and repeat the entire analysis cycle.

To automate the ranging function of the polarograph, a decade resistance circuit board was constructed and installed inside the Model 264A. Figure 5-7 is a wiring diagram of this circuit with component descriptions and connector pin functions presented in Tables 5-7 and 5-8, respectively. When activated, this circuit bypasses the Model 264A current range circuit and provides for the correct feedback resistance as controlled by the instrument's software. Similar to the run/stop circuit, activation of the electromechanical relays on the decade resistance board is provided by 5 VDC command signals from the Kiethley PCM3 circuit board. When the decade resistance board is activated, manually changing the current ranging switch on the 264A front panel has no effect on the amplifier gain.

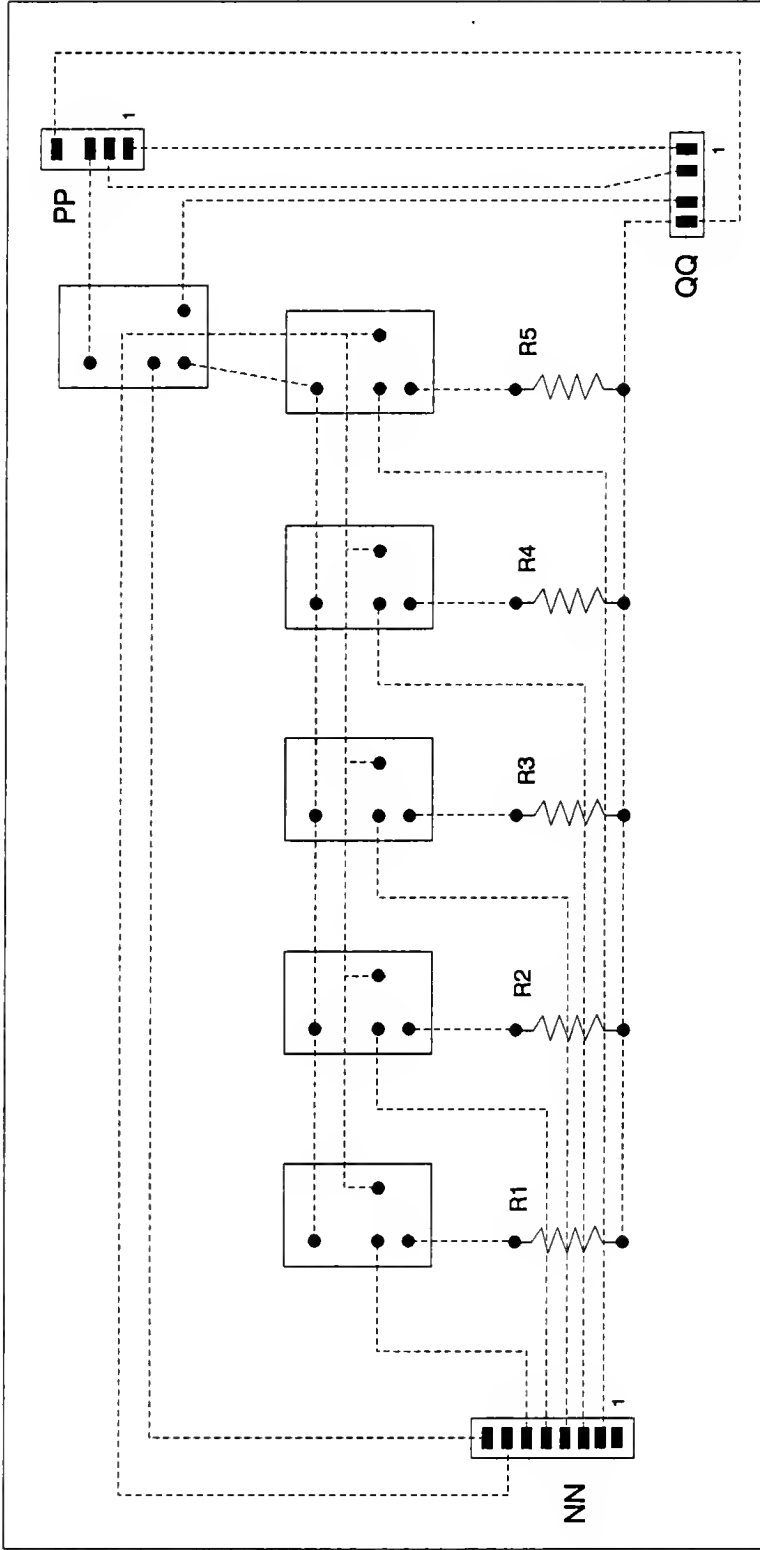


Figure 5-7. Schematic of printed circuit board which provides decade resistance ranging functions. Table 5-7 provides component descriptions and Table 5-8 lists connector pin functions.

Table 5-7
Component Listing and Description of Decade Resistance Board

<u>Component</u>	<u>Description</u>
Electromechanical Relays	5 VDC, 72mA, SPDT 2A at 125 VAC Radio Shack Type 275-243
Connector NN	8 pin Post Header, Friction Lock Amphenol Type 1-640456-0
Connector PP	5 pin Post Header, Friction Lock Amphenol Type 640456-6 Attaches to J6 on 264A Main Board
Connector QQ	5 pin Post Header, Friction Lock Amphenol Type 640456-6 Attaches to 264A Current Range Board
Resistor R1	2M ohm, 1%, 0.5 W
Resistor R2	200k ohm, 1%, 0.5 W
Resistor R3	20k ohm, 1%, 0.5 W
Resistor R4	2k ohm, 1%, 0.5 W
Resistor R5	200 ohm, 1%, 0.5 W

Table 5-8
Decade Resistance Board Connector Pin Functions

<u>Pin#</u>	<u>Current Range</u>	<u>Function</u>
NN-1	--	Not Connected
NN-2	00nA	Activates Relay 5
NN-3	μ A	Activates Relay 4
NN-4	0 μ A	Activates Relay 3
NN-5	00 μ A	Activates Relay 2
NN-6	mA	Activates Relay 1

Figure 5-8 shows the recommended locations for the run/stop, PCM3, and decade resistance circuit boards which have been installed within the Model 264A control unit. Communication between the PCM3 and the data acquisition and control system is provided by a 50 pin ribbon cable with appropriate connectors installed in the rear panel of the Model 264A and the Kiethley 575 system. Component listings and descriptions for these connectors are presented in Tables 5-9 and 5-10.

Description of Digital Logic Circuit

Communication between the Model 303A polarograph and the Model 264A control unit takes place through a 25 pin D-sub ribbon cable (EG&G Model C0113). In addition to providing power to the Model 303A, the control unit sends digital command signals to activate the polarograph's functions of stir, mercury drop dispense/dislodge, and nitrogen purge. Polarographic scan results are returned the control unit through this same cable.

In the Model 303A, mercury flows from the mercury reservoir through a thin glass capillary (0.006 inch diameter) to form a drop at the capillary tip. During the electrochemical reaction, the mercury column within the capillary carries the resulting redox current from the mercury drop to the electrical output circuit. If a discontinuity should occur in the mercury column, however, no electrical potential can be applied to the drop and the electrochemical reaction cannot take place. In this event, reestablishing the mercury column continuity

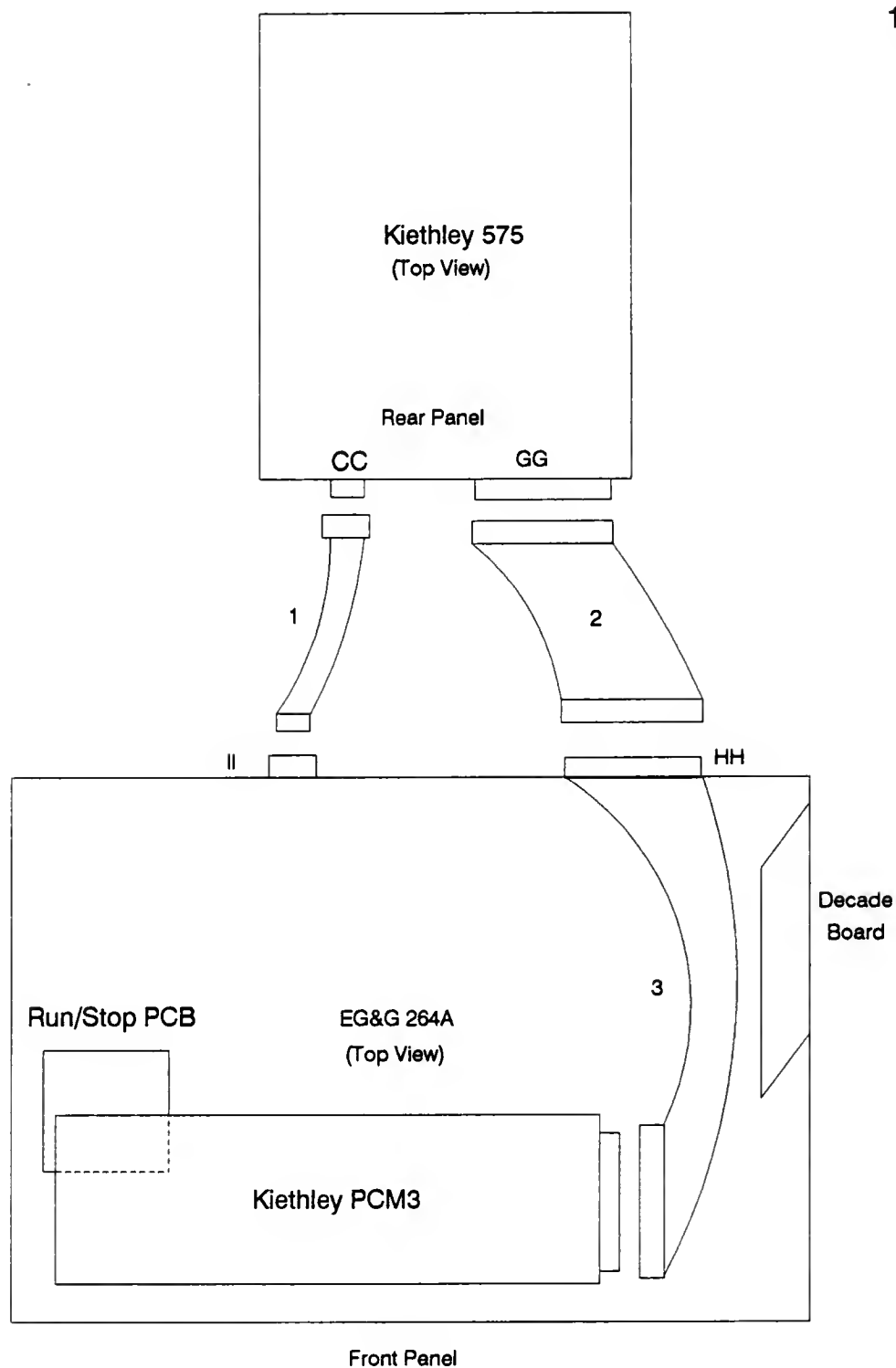


Figure 5-8. Schematic of Model 264A showing recommended locations for auxiliary circuit boards. Component descriptions are listed in Table 5-9. Connector pin functions are provided in Table 5-10.

Table 5-9
Component Listing and Description of Model 264A

<u>Figure 5-8 Code</u>	<u>Function</u>	<u>Description</u>
1	Connects CC to II	9 pin D-Sub ribbon cable with 3M type plug 8209-6000 and socket type 8309-6000
2	Connects GG to HH	50 pin delta ribbon cable with 3M F card edge connector type 3415-0001 and 3M delta plug type 3564-1000
3	Connects Kiethley 575 to PCM3	50 pin delta ribbon cable with 3M socket type 3565-1000 and 3M F card edge connector type 3415-0001

Table 5-10
Model 264A Connector Pin Descriptions

<u>Pin#</u>	<u>Color</u>	<u>575 Channel</u>	<u>264A Connection</u>	<u>Function</u>
II-1	PUR	ANIN1	DPM-6	Reads I "High" in I vs. V scan
II-2	BLU	ANIN1	DPM-H	Reads I "Low" in I vs. V scan
II-3	RED	ANIN4	J14-5	Reads scan status from scan LED
II-4	GRY	ANIN3	I/E monitor rear panel	Reads V "High" in I vs. V scan
II-5	GRN	ANIN3	GND	Reads V "Low" in I vs. V scan

requires repeated manual activation of the drop dislodge/dispense function switches (located on the front panel of the polarograph) until continuity is regained. Disruption of the mercury column continuity occurs primarily during movement or excessive vibration of the polarograph. Since the lead-in-air monitor was intended for portable use, it was anticipated that loss of mercury column continuity would occasionally take place.

To eliminate the need for manual control, a circuit board was designed to provide to automatically trigger these dispense/dislodge function switches. In the Model 303A, a digital TTL logic low signal applied to pin 10 from the Model 264A control unit triggers a single dislodge/dispense sequence. Figure 5-9 is a schematic of the circuit board designed to provide for external application of this signal without interfering with the normal operation of the polarograph. Component descriptions and connector pin functions are provided in Tables 5-11 and 5-12. The combination of switching diodes and resistors shown for pin 10 represents a standard digital AND logic gate. The circuit shown allows a separate digital low TTL signal to be applied when desired by the data acquisition and control system. Experiments have shown that initiating the drop dislodge/dispense function through fifteen cycles is sufficient to reestablish column continuity.

To enable stirring of the flow through polarographic cell during the rinse cycle, a similar logic gate on the circuit board is applied to pin 8 of the ribbon cable. External control of the polarograph's nitrogen purge function is provided

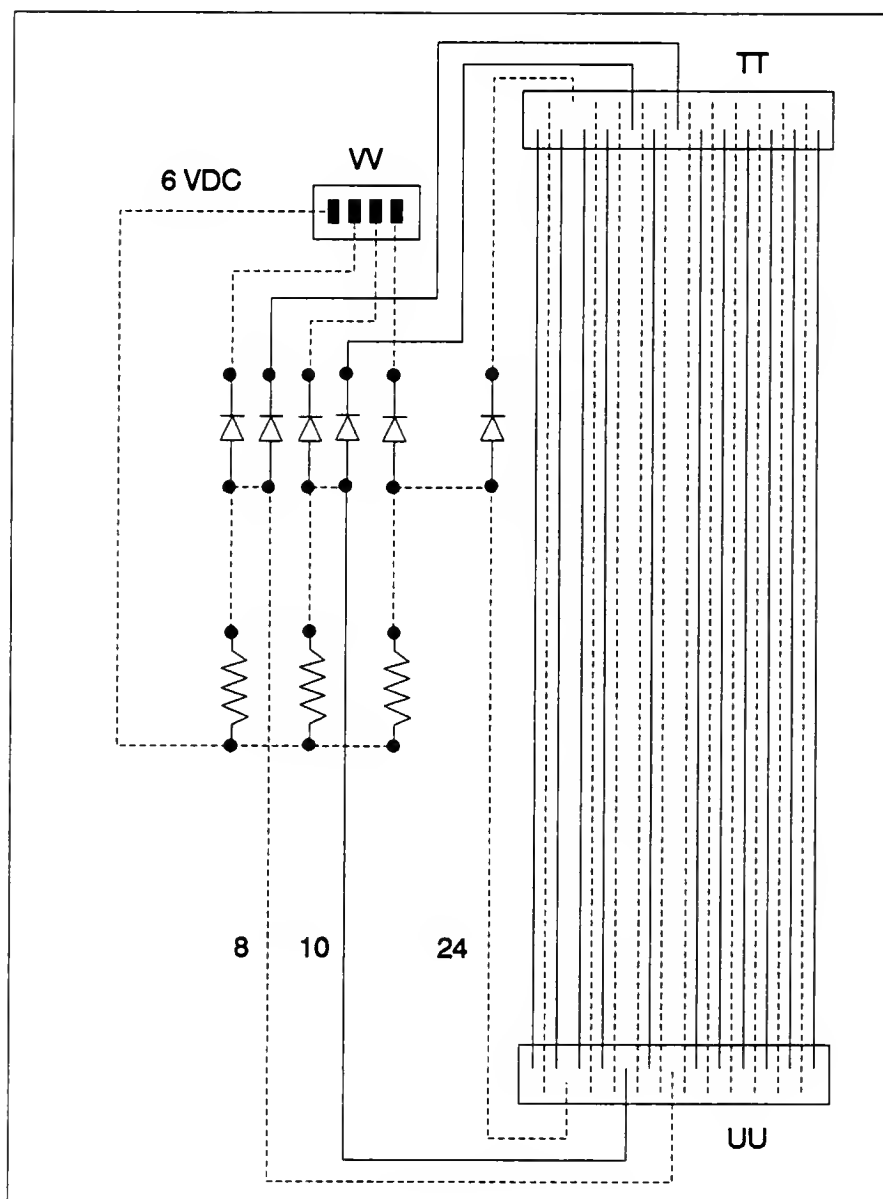


Figure 5-9. Schematic of printed circuit board which provides logical switching of stir, purge, and drop dispense/dislodge functions. Component descriptions are listed in Table 5-11 and connector pin functions listed in Table 5-12.

Table 5-11
Component Listing and Description of Digital Logic Board

<u>Component</u>	<u>Description</u>
Connector TT	Right Angle D-Sub Header Plug Dupont Type 71563-325
Connector UU	Right Angle D-Sub Header Socket Dupont Type 71564-325
Connector VV	4 pin Post Header, Friction Lock Amphenol Type 640456-4
Switching Diodes	Silicon diode, 1N914 Radio Shack Type 276-1122
Resistors	Metal film, 1k ohm, 1%, 0.5 W Dale Type RN-65D

Table 5-12
Digital Logic Board Connector Pin Descriptions

<u>Pin#</u>	<u>Color</u>	<u>575 Channel</u>	<u>Module 1 Connection</u>	<u>Function</u>
VV-1	YEL	ANOUT1	BB-9	Supplies 6 VDC
VV-2	ORG	DIGOUT6	BB-11	Initiates STIR
VV-3	BLK	DIGOUT7	BB-12	Initiates Hg Drop Dislodge/Dispense
VV-4	PNK	DIGOUT5	BB-10	Initiates Purge

by control of pin 24. No modifications were made to the ribbon cable's other pin positions.

Measurement of Analysis Results

In polarography, test results are analyzed on the basis of measured redox current versus the applied scan voltage. Resulting peak heights are proportional to concentration while peak positions identify the component undergoing the redox reaction. When using the Model 264A control unit, this information is typically transmitted to an x-y recorder and the resulting polarogram is interpreted by the user. As an alternative to use of an x-y recorder, either measured redox current (converted to voltage) or scan voltage can be selectively displayed on a 2 VDC digital panel meter (DPM) located on the front of the 264A. The user may then manually record and interpret the results displayed during the analysis. Obviously, neither of these output options lend themselves to convenient automation.

To monitor the redox current produced during a scan, connections were made to the appropriate pins of the Model 264A panel meter (see Table 5-10) and signals transmitted to an analog input channel on the data acquisition and control system through a 9 pin ribbon cable. The scan voltage is monitored by connection to the electrometer monitor on the rear panel of the 264A, transmitted through the ribbon cable, and analyzed on a separate analog input channel.

Monitoring these analog signals does not take place continually but only during the time of the analysis scan (stripping phase). On the Model 264A, the onset of the scan is signaled to the user by powering a light emitting diode (LED) located on the unit's front panel. Monitoring the voltage drop across this LED provides a convenient means of detecting when a scan has begun. This signal thus acts as a hardware trigger to notify the lead-in-air monitor's software to begin recording the scan results. When the LED is deactivated, recording of the scan is terminated and the recorded data is analyzed.

CHAPTER 6

DESCRIPTION OF FLUID HANDLING SYSTEM

Design of the lead-in-air monitor's particle collection and lead analysis section were based on the use of a liquid collection substrate. Specifically, lead-based aerosols are deposited in 50 ml of 0.5M nitric acid with a 200 ppb copper internal standard. Use of liquid substrates dictates the need for a liquid storage system, an accurate and repeatable liquid delivery system, and a waste disposal system. In addition, the static type cell of the commercially available polarograph must be replaced with a flow-through type cell. All system components must be compatible with the acidic solution and not contaminate the solution. Ideally, the liquid handling system would represent a closed system thus minimizing possible contamination from the outside environment. In this section, the details of the liquid handling system will be discussed.

Figure 6-1 is a schematic of the liquid handling system that was developed based on the monitor's requirements. Component descriptions are presented in Table 6-1. To minimize fluid contamination, all tubing and compression fittings used in the liquid handling system are composed of teflon. The nitric acid electrolyte is stored in a 5 liter capacity teflon reservoir. To ensure that the existing electrolyte supply is sufficient for a sampling series, a

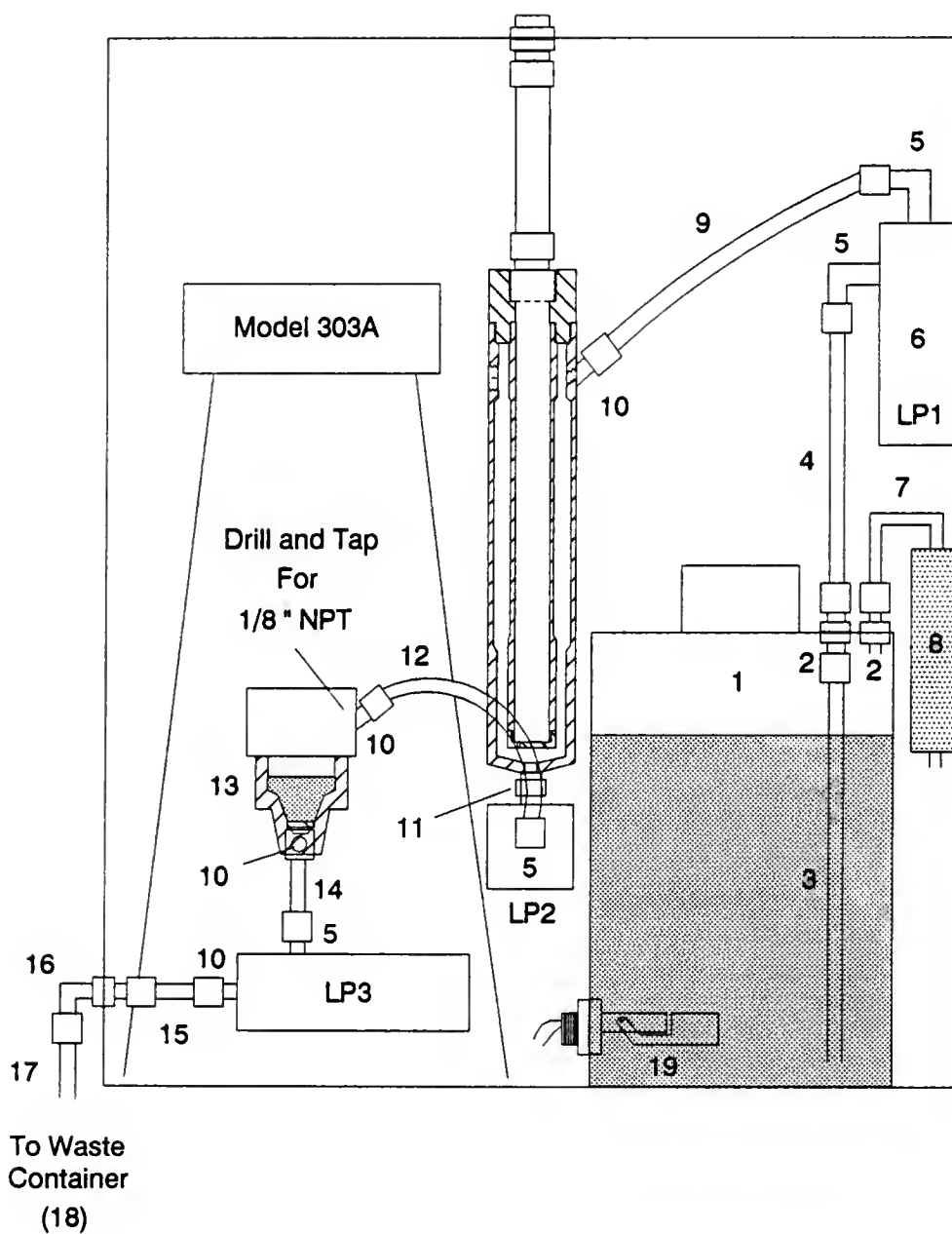


Figure 6-1. Schematic of Module 1 liquid handling system.

Table 6-1
Listing and Description of Liquid Handling Components

<u>Figure 6-1 Code</u>	<u>Description</u>
1	Teflon Bottle, 5 Liter Capacity Fisher Scientific, Model 02-892-50H
2	Teflon Bulkhead Union, 1/4 in. Tube Cole Parmer, Model N-06482-34
3	Teflon 1/4 in. Tube, 8 1/2 in. Cole Parmer, Model N-06375-02
4	Teflon 1/4 in. Tube, 7 7/8 in.
5	Teflon Male Adaptor Elbow, 1/4 in. Tube to 1/8 in. NPT(M) Cole Parmer, Model N-06374-12
6	Liquid Pump, Positive Displacement 120 VAC, 14.2W Valcor Scientific, Model SV602C115PV
7	Nalgene Tube, 5/16 in. O.D., 3/16 in. I.D., 9 in. Fisher Scientific, Model 14-176-14
8	Drying Tube, 4 in. with Glass Wool Fisher Scientific, Model 09242A
9	Teflon 1/4 in. Tube, 12 1/2 in.
10	Teflon Male Adaptor, 1/4 in. Tube to 1/8 in. NPT(M) Cole Parmer, Model N-06374-02
11	Teflon Nipple, 1/8 in. NPT(M) Cole Parmer, Model 06483-00

Table 6-1--continued

<u>Figure 6-1 Code</u>	<u>Description</u>
12	Teflon 1/4 in. Tube, 12 in.
13	Teflon Flow Through Cell Machine to Specifications
14	Teflon 1/4 in. Tube, 5 in.
15	Teflon 1/4 in. Tube, 2 3/8 in.
16	Teflon Elbow Panel Mount Cole Parmer, Model N-06379-69
17	Teflon 1/4 in. Tube, 22 in.
18	Nalgene Polypropylene WM Bottle 10 liter Capacity Fisher Scientific, Model 02-961-65A
19	Polysulfone Level Switch Gems Services, Model A76147

polysulfone fluid level switch has been installed near the base of the reservoir. The status of the level switch is monitored through the data acquisition and control system. At the top of the reservoir, two bulkhead tube fittings have been installed to provide for the fluid transfer and corresponding vent makeup air. To avoid possible contamination of the reservoir, this makeup air is filtered through a drying tube packed with glass wool.

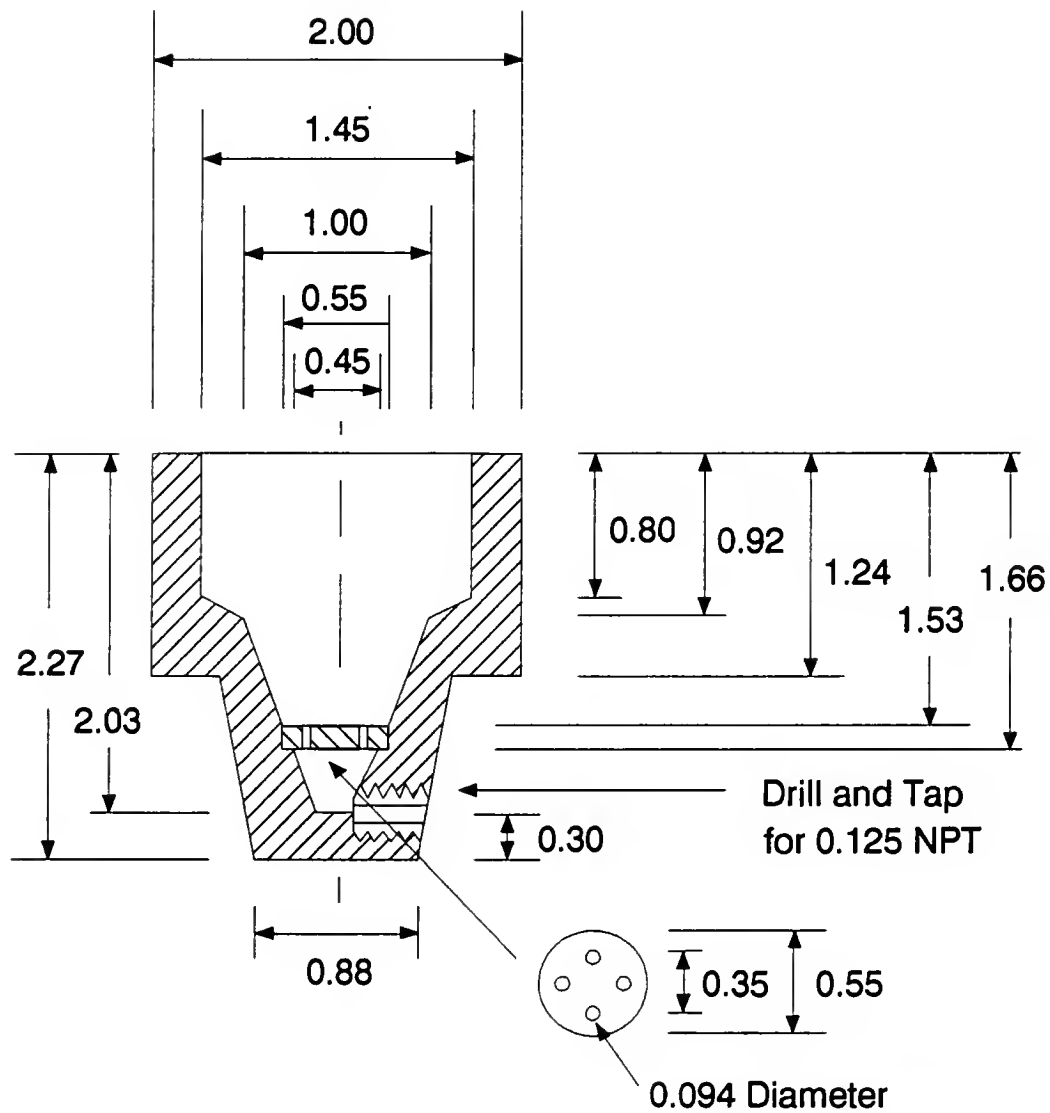
Careful consideration was given to selection of the system's fluid handling pumps. Since lead mass is estimated from the product of the measured lead concentration and the liquid volume, the exact liquid volume dispensed to the impactor must be known. A liquid pump of selectable delivery rate and possessing high repeatability was required. The pumps must be self-priming, be compatible with the acidic solution, meet cost requirements, and be adaptable to computer-based power control.

Given these considerations, three identical fluid metering pumps were purchased (Valcor Scientific, Model SV602C115PV) and incorporated into the system. These pumps are solenoid operated, positive displacement types with polypropylene and glass wetted surfaces. Application of 115 VAC power to the solenoid coil provides for one-half of the pumping cycle and de-energization of the coil completes the pumping cycle. The pump may be cycled up to 120 cycles per minute. Digital signals to solid-state power relays establish the pumping rate. In addition, the pump's piston stroke length can be selected using a manual adjustment screw. Thus, the overall fluid delivery rate (up to

120 ml/min) can be controlled by proper combination of the piston stroke length and the rate of the applied power cycle. The repeatability of these pumps has been measured to be within 2%.

Once 50 ml of the electrolyte is dispensed to the impactor and the air sampling run completed, the solution is transferred to the polarograph for analysis. In the commercial polarograph, 10 to 15 ml of the solution to be analyzed is normally placed in a static cup and a teflon coated stirring bar placed in the cup. The cup is then installed in the polarograph to immerse the electrodes in the solution. A spring-loaded metal support is then positioned to support the cell. To provide for solution stirring during the deposition step, an EG&G Model 305 stirrer is placed under the cell. After each separate analysis, the cup must be removed, cleaned, filled with a new solution and the installation and analysis steps repeated. Since these steps would be difficult to automate, a flow-through cell was designed and machined to replace this static cell arrangement.

Figure 6-2 is a detail drawing of this flow-through cell. The unit is cylindrical in cross-section and was machined from teflon. A separate 0.55 inch diameter disk was machined and is press-fit within the neck of the cell and supports a star-shaped stirring bar (Fisher Scientific, Model 14-511-96A). The top section of the cell was machined to seal against the o-ring of the polarograph's electrode section when the cell is supported by the Model 305 stirrer.



Title	Part No.	Material
Flow-through Cell	DRW-6	Teflon
Date	Scale	
Aug. 30, 1991	Approximate	
R.W. Vanderpool	Dimensions are in inches. Tolerances plus or minus 0.01 inches unless otherwise noted	

Figure 6-2. Detail drawing of flow-through cell designed for the polarograph.

Once installed, the flow-through cell need not be removed except during periodic maintenance of the electrodes. Fluid is introduced into the cell through the polarograph's plastic electrode support block which has been drilled and tapped to accept a male tubing adaptor. Since the capacity of the flow-through cell is approximately 22 ml, the entire 50 ml contents of the impactor cannot be totally transferred to the cell. Therefore, approximately 35 ml is rinsed through the cell by simultaneous operation of liquid pumps 2 and 3. Liquid pump 3 is then deactivated and the remaining 15 ml fills the cell for analysis. Since the polarograph measures lead concentration rather than absolute lead mass, the exact volume of solution analyzed is not critical.

Once the analysis is completed, the liquid handling undergoes a complete acid rinse cycle using 50 ml of the electrolyte. This includes a 10 second air sampling period to thoroughly rinse the wetted surfaces of the teflon impactor. Fifteen milliliters of the electrolyte remains in the flow-through cell to ensure that the reference electrode does not dry out. Prior to the next sampling and analysis test, this entire rinse cycle is automatically repeated. Rinsing the liquid components twice between sampling runs has proved to be effective in minimizing contamination from one test run to another. Because each sampling run includes two rinse cycles, 150 ml of electrolyte is consumed for each test run. The 5 liter container thus has sufficient capacity for approximately 30 runs before a low level condition is indicated by the reservoir's fluid level switch.

Recall that nitrogen purging of the cell's contents is required to remove the electroactive influence of any dissolved oxygen. Figure 6-3 is a schematic of the nitrogen handling system with component descriptions provided in Table 6-2. Nitrogen is supplied from a disposable cylinder with a capacity of 34 liters at standard temperature and pressure. A 0-500 psig gauge at the cylinder outlet allows inspection of the cylinder's current nitrogen supply. A regulator at the cylinder exit provides a nitrogen output pressure of 20 psig. The gas then passes through a normally-closed 115 VAC activated solenoid valve. Although the polarograph itself contains a nitrogen flow solenoid valve, it merely directs the nitrogen gas either through the cell contents or over the liquid surface. Without the additional solenoid to supply the nitrogen only when needed, the limited nitrogen supply would soon become depleted.

Nitrogen flow through the polarograph is adjusted and monitored using a 0 to 100 cc/min capacity rotameter. Although the actual flowrate is not critical, the nitrogen flowrate should be adjusted to be between 30 and 50 cc/min. Once the rotameter is set, it does not normally require readjustment until the nitrogen cylinder need be replaced. Based on the nitrogen purge requirements of each analysis cycle, the nitrogen cylinder need be replaced only after approximately 200 separate analyses.

Figure 6-4 is a wiring diagram of the components associated with the nitrogen handling system. Component descriptions and sources are listed in Table 6-3. Connector pin descriptions are presented in Table 6-4.

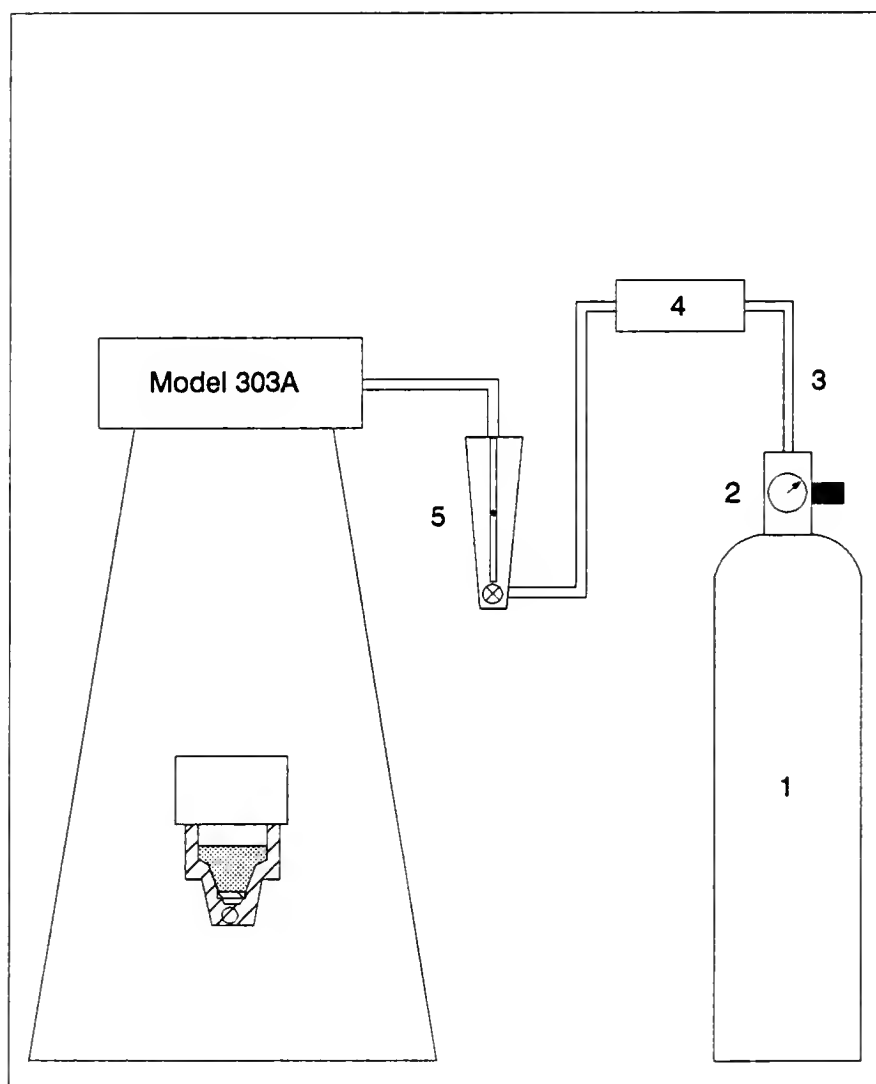


Figure 6-3. Schematic of Module 1 nitrogen handling system.

Table 6-2
Listing and Description of Nitrogen Handling Components

<u>Figure 6-3 Code</u>	<u>Description</u>
1	Ultra-High Purity Nitrogen Certified >99.999 mole % 500 psig, 34 liters at 70 °F Alphagaz, Cylinder Size 7D
2	Regulator, Delivery Pressure = 20 psig with 0-500 psig Guage, CGA 600 Outlet Alphagaz, Model 713
3	Norprene Tubing, 5/16 in. O.D., 3/16 in I.D. Fisher Scientific, Model 14-169-25D
4	Delta Solenoid Valve with Teflon Wetted Parts, 115 VAC, 0.15A, 1/8 in. Ports 2-Way Normally Closed Fluorocarbon, Model DV2-122NCA1
5	Rotameter, Brooks Instr. Co., Model 1350 with R-2-65A Tube and Sapphire Float. Flush Panel Mounted.

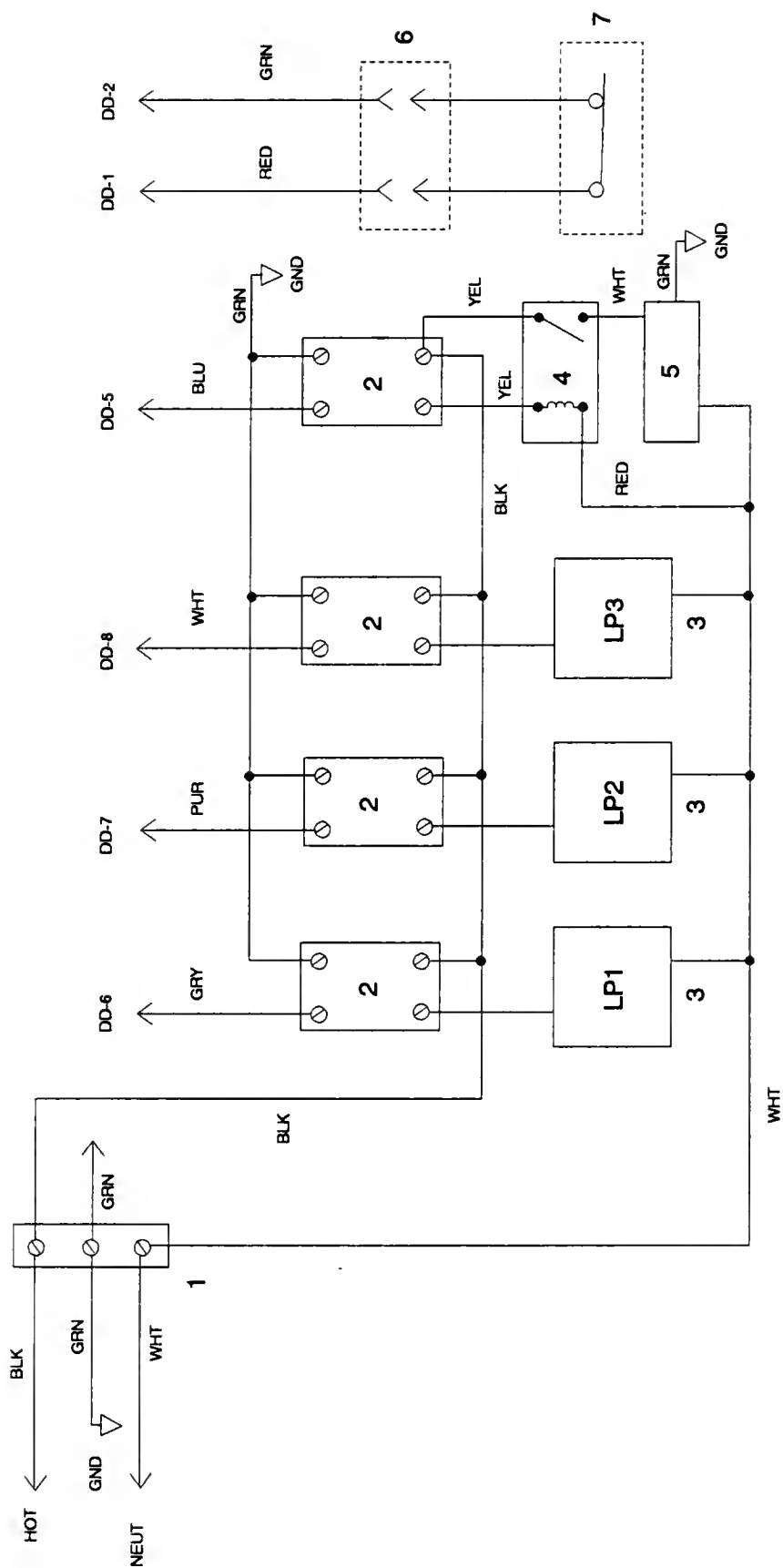


Figure 6-4. Wiring diagram of Module 1 electrical components.

Table 6-3
Module 1 Electrical Component Listing and Description

<u>Figure 6-4 Code</u>	<u>Description</u>
1	Barrier Terminal Block 3 Contact, Double Row, 20A Cinch, Inc. Type 3-141
2	Solid State Relay, 240 VAC/25A 3-32 VDC Input Potter and Brumfield Type SSR-240D25
3	Liquid Pumps, 125 VAC Valcor Scientific Type SV602C115PV
4	Electromechanical Relay, 125 VAC 15mA, DPDT, 10A at 125 VAC Radio Shack Type 275-217
5	Delta Solenoid Valve with Teflon Wetted Parts, 115 VAC, 0.15A, 1/8 inch Ports 2-Way Normally Closed Fluorocarbon, Model DV2-122NCA-1
6	Quick Connector Amphenol 2 Circuit Pin Housing, type 1-480319-0 2 Circuit Pin Socket, type 1-480318-0 Contacts, 24-18 AWG, types 60618-1 and 60617-1
7	Polysulfone Fluid Level Switch Gems Sensor Division Model A76147

Table 6-4
Module 1 Electrical Connector Pin Descriptions

<u>Pin</u>	<u>Color</u>	<u>575 Channel</u>	<u>Function</u>
BB-1	RED	DIGOUT0	Powers Fluid Level Switch
BB-2	GRN	ANIN2	Reads Fluid Level Switch
BB-4	GRN	GROUND	Ground
BB-5	BLU	DIGOUT1	Activates Nitrogen Solenoid
BB-6	GRY	DIGOUT2	Activates LP1 for Impactor Fill
BB-7	PUR	DIGOUT3	Activates LP2 for Impactor Empty
BB-8	WHT	DIGOUT4	Activates LP3 for Cell Empty
BB-9	YEL	ANOUT0	6 VDC to Decade Board
BB-10	PNK	DIGOUT5	Initiates Purge Function
BB-11	ORG	DIGOUT6	Initiates Stir Function
BB-12	BLK	DIGOUT7	Initiates Drop Dislodge/Dispense

CHAPTER 7

PROGRAM DESCRIPTION AND OPERATION

In the lead-in-air monitor, automated sequencing of the system's components takes place using a Kiethley 575 data acquisition and control system interfaced with a PC-Brand 286/25 IBM-compatible computer. Interaction between the computer and the data acquisition and control system takes place through a Kiethley data acquisition interface card which was installed inside a vacant 8 bit slot within the computer. The KDAC500 software package was then installed on the computer's 20 megabyte hard drive.

In the Kiethley 575 system, the user can access the various input/output module channels using the Kiethley software linked with a GW-Basic programming environment. Installation of the KDAC500 software requires the user to specify configuration options which include channel input/output names for each module channel. Individual channels are accessed through machine language call statements which use channel names as arguments. A listing of the names chosen for the lead-in-air monitor is presented in Table C-1 in Appendix C. For a complete listing of the configuration settings, the user may view the contents of the CONFIG.TBL file located on the computer's hard drive.

Operation of the lead-in-air monitor is controlled through a single GW-Basic program, "MAIN", which has been written for this purpose. This program

was installed on the hard drive and automatically loads and executes when the computer is powered up. A complete listing of this program is provided in Appendix D.

Program execution begins by initializing program variables (such as standard pressure, nitrogen purge time, etc.), dimensioning basic arrays, and initializing the digital logic board to deactivate purge, stir, and dislodge/dispense functions. The user is then presented with the main program menu of operating options. This allows selection of either single or continuous sampling analysis, analysis of contents of polarographic cell, various fluid handling options, view or print of previous saved results, or an option to exit the program.

The single sampling and analysis option is chosen if the user wishes to perform single point analysis at various sampling locations within the battery plant. The unattended sampling and analysis option allows the user to perform multiple sampling and analyses tests at the same sampling location. In either case, the user is first prompted to enter pertinent sampling conditions at the desired point of sampling. These inputs include measured stack gas temperature and stack gas velocity pressure which are necessary to calculate the stack gas velocity using the equation

$$V = K C_p \sqrt{V_p} \sqrt{\frac{T}{P_s M}} \quad (7-1)$$

where V is the stack gas velocity, K the appropriate units constant, C_p is the pitot tube correction factor, V_p is the measured velocity pressure, and T, P_s , and

M are the stack gas temperature, static pressure, and molecular weight, respectively. In the program, values of stack gas pressure and molecular weight are assumed for standard air. The value of the pitot tube correction factor (0.99) assumes use of a standard pitot tube for velocity pressure measurement. If needed, the appropriate program lines may be rewritten to modify these assumed values.

Based on the calculated velocity, the program computes the ideal nozzle diameter necessary to achieve isokinetic sampling. The user is then prompted to enter the diameter of an available nozzle close to this ideal size. If this selected diameter requires a flowrate outside the sampling system's 10 to 30 liters per minute design range, the user is prompted to modify the selection. If static (still air) sampling is to be performed, the user is prompted to enter a zero value for the velocity pressure. A fixed 15 actual liters per minute sampling flowrate is used for all static sampling tests.

Prior to the air sampling, a complete rinse of the liquid handling components is performed by proper sequencing of the liquid handling pumps. The status of the electrolyte level switch is also checked at this time to ensure that the available electrolyte supply is sufficient for a complete sampling cycle. Once the rinse is completed, 50 ml of the electrolyte is transferred to the impactor.

Based on the required nozzle volumetric flowrate, the mass flowrate required by the mass flow controller is calculated. The appropriate analog

command voltage is signaled to the mass flow controller to set the position of the solenoid valve. A one volt command signal is equivalent to 10 liters per minute at 32 °F. The air pump is then activated and sampling is performed for 15 minutes unless the test is manually aborted by the user. At ten second intervals during the sampling, the measured sampling flowrate is monitored from the mass flow controller output and this value stored in an array created for this purpose. At the end of the sampling period, the air pump is turned off and the average flowrate computed from the array of measured values.

Once the air sampling is completed, the liquid solution is transferred from the impactor to the polarograph cell for lead analysis. The cell's contents are first purged with nitrogen by activating the normally-closed solenoid for three minutes. During the purge cycle, activation of the STIR function on the digital logic board provides for stirring of the solution. The program then activates the RUN function on the RUN/STOP circuit board to initiate a polarographic analysis. Following the required deposition step to deposit the aqueous lead onto the surface of the electrode, the stripping step is initiated to quantify the lead concentration. Once this scan is detected, the resulting current versus voltage readings are recorded every 0.2 seconds and stored in separate arrays. Typically, a full polarographic scan requires 80 seconds to complete. When the scan is completed, the readings are terminated and the current and voltage arrays analyzed.

In polarography, the position of the scan peaks identifies the analyte present while its peak height is proportional to its concentration in solution. In the lead-in-air monitor, two separate primary peaks will be observed, one corresponding to the collected lead and the other corresponding to the internal copper standard. The peak heights are determined by examining the contents of the current and voltage arrays. As discussed, the ratios of the peak heights rather than their absolute heights are used to quantify the lead concentration in solution.

Prior to the analysis, the decade resistance board sets the scan range to the value appropriate for lead concentrations expected during occupational or recirculation air monitoring. In this range, the response to the 200 ppb copper internal standard is typically about 1.6 volts but can vary by approximately 50%. If the response is less than 0.3 volts, however, it signals that either the electrolyte was improperly prepared or that a discontinuity exists in the mercury column. In this event, the digital logic board is activated to initiate fifteen mercury drop dispense/dislodge cycles to reestablish the mercury column continuity. The lead analysis scan is then repeated. If the problem persists, the operator is prompted to correct the problem.

Alternatively, if the actual lead concentration is higher than approximately 2000 ppb, an overrange condition will occur and the decade board will automatically reduce the output amplifier gain by a factor of ten. The analysis will then be repeated.

Once the solution's lead concentration is quantified, the lead-in-air concentration is calculated from the average flowrate, sampling time, liquid lead concentration, and solution volume. Reported lead concentration units of micrograms per cubic meter are based on a standard temperature of 25 °F.

A summary of the analytical results is presented on the video display for the user's inspection. Reported are values of liquid and air lead concentration, air volume sampled, height of lead and copper peaks, and value of the percent isokinetic during the sampling run.

Whether operating in single point sampling mode or unattended operation, the test results are then automatically stored on the computer's hard drive for later inspection. Rather than requiring the user to enter a separate filename for each test run, the program reads the computer's internal clock and uses the current date as the filename. For successive runs performed on the same test date, the program simply adds the new test results to the same random access file. The information saved for each test run includes the test's starting time, test duration, average flowrate, liquid and air lead concentrations, and a test run description. This description is entered by the user at the start of the sampling run and may contain up to 30 characters.

Following storage of the test results, the liquid handling system then undergoes a complete rinse cycle. If single point sampling had been performed, the user is returned to the main program menu. If the instrument is currently operating in unattended sampling mode, the complete sampling and

analysis cycle is repeated. Unattended operation will continue until manually aborted or until the electrolyte supply is insufficient for continued operation. In the latter event, the user is prompted to refill the reservoir to allow for continued operation.

In the main menu, a fluid handling option exists which presents the user with a separate menu of various fluid handling options. The user is able to either perform a main system rinse cycle or operate any of the three liquid pumps to either fill the impactor, empty the impactor, or empty the polarographic cell. These latter options are useful if the user wishes to periodically verify the calibration of the liquid handling pumps. Manual control of the sampling pumps is also useful should a power outage occur and interrupt proper sequencing of the fluid handling components.

A separate main menu option enables analysis of the polarographic cell contents without any air sampling. This option can be used to verify the polarograph's response to the internal copper standard. Alternatively, this option can be used to check the analysis accuracy of a lead standard sample introduced into the cell.

The final main menu option enable the user the view or print previously saved test results. All tests results are stored in the subdirectory "C:\K500\RESULTS" created for this purpose. Using a separate menu, the user can request a listing of existing file names. The contents of a particular file may either be viewed on the computer's monitor or a hard copy listing of the

file made. Note that printing previous test results requires the user to connect the computer to a parallel dot-matrix or letter quality printer.

CHAPTER 8 FIELD EVALUATION OF PROTOTYPE MONITOR

Introduction

Accurate evaluation of lead-in-air concentrations requires accurate air sampling, particle transport and collection, and lead analysis. In previous sections, the details of each of these instrument subsystems has been described in detail. Laboratory evaluation of these subsystems suggested that they each performed approximately as designed. In order to fully evaluate the capabilities of the complete instrument, however, field tests were conducted in a battery plant under conditions of the instrument's intended use. Although the primary intent of the field tests was to evaluate the ability of the monitor to quantify lead-in-air concentrations, the field tests were also designed to evaluate the instrument's overall ruggedness, portability, and the extent to which unattended operation could be expected.

Experimental Methods

Prior to the field tests, all field sampling equipment and sample containers were prewashed with 6M nitric acid, rinsed in D.I. water, and air dried. Individual collection vials, filter holders, and sampling nozzles were

sealed in separate press-lock style plastic bags. Glass fiber filters to be used for the field tests were preheated at 300 °F for two hours then stored in a desiccator until the time of their use. Three of these filters were later selected at random and subjected to extraction procedures to determine their background lead content. As measured by GFAA, the lead content of these filters was found to be insignificant compared to the lead mass collected during the field tests.

As quality control checks, a series of lead standards was prepared in the laboratory prior to the field tests. These samples consisted of 0, 100, 200, and 300 ppb lead standards prepared in 0.5M nitric acid with a 200 ppb internal copper standard. All samples were stored in prewashed polypropylene containers. A separate container of the 100 ppb lead standard was prepared and was used periodically in the field to verify the accuracy of the monitor's analytical section. One set of the 0 to 300 ppb lead standards was stored in the laboratory at 4 °C and was used as laboratory standards to check the long-term stability of lead solutions stored in this manner. A second set of these 0 to 300 ppb standards was used as field control standards which were transported to the field site but not opened during the week of testing. Use of field standards was designed to check for any inadvertent container contamination which might occur at the testing site.

Field evaluation of the lead-in-air monitor occurred during the week of July 15-19, 1991 at a battery plant in the southeast United States. All sampling

equipment preparation and storage took place in a quality control laboratory within the battery plant. The possibility of sample contamination by trace quantities of lead required the use of an area as isolated from the battery production process as possible. Sample contamination within this quality control laboratory was expected to be minimal.

Upon arrival at the battery plant, an initial check of the monitor's condition revealed two key components were inoperable. First, the nitrogen solenoid valve did not respond when control power was applied. This solenoid was replaced in the field with a spare unit. The second malfunction encountered involved the liquid metering pump which dispenses electrolyte to the impactor prior to air sampling. Inspection of the unit revealed that the pump's piston would not move when the pump's coil was activated. As a result, no dispensing of the electrolyte could occur. Once the pump was disassembled, its components cleaned, and reassembled, the problem did not reoccur during the week of field tests. No additional operating problems with the lead-in-air monitor were encountered during the remainder of the week's testing.

The majority of the field tests were performed at the process site where the dried, pasted grids are manually off-loaded from the conveyor belt and stacked onto platens for later transport to the curing ovens. This process site was chosen because it represented a site of aerosol production similar to that expected in recirculation air systems in terms of the aerosol's size distribution

and concentration. Unlike some other potential sampling sites, this site allowed sampling without interfering with the activities of the plant personnel. Figure 8-1 is a photograph of the approximate position of the lead-in-air monitor in relation to the plate off-bearing process.

In order to determine the accuracy of the lead-in-air monitor, collocated total filter sampling was performed during the majority of the field tests. As shown in Figure 8-2, an aluminum filter holder was positioned next to the monitor's sampling inlet. The filter holder has a circular inlet with a diameter of 1.375 inches. Glass fiber filters of 47 mm diameter (Gelman, A/E 61631) were used for these tests and display essentially 100% collection efficiency independent of particle size or flowrate. The filters were loaded and unloaded in the battery plant laboratory and the filter holder covered with a plastic bag until just prior to sampling. During the week of testing, three blank filters were separately loaded and unloaded into the filter holder without any air sampling in order to measure possible contamination during the filter handling. Subsequent lead extraction and analysis of these filters showed that total filter contamination was not significant.

Flowrate through the total filter was provided by connection to an EPA Method 5 sampling box which allowed control of the flowrate and measured the total volume of air sampled during the sampling run. For the static sampling tests, the total filter flowrate was adjusted to equal that of the lead-in-air monitor.

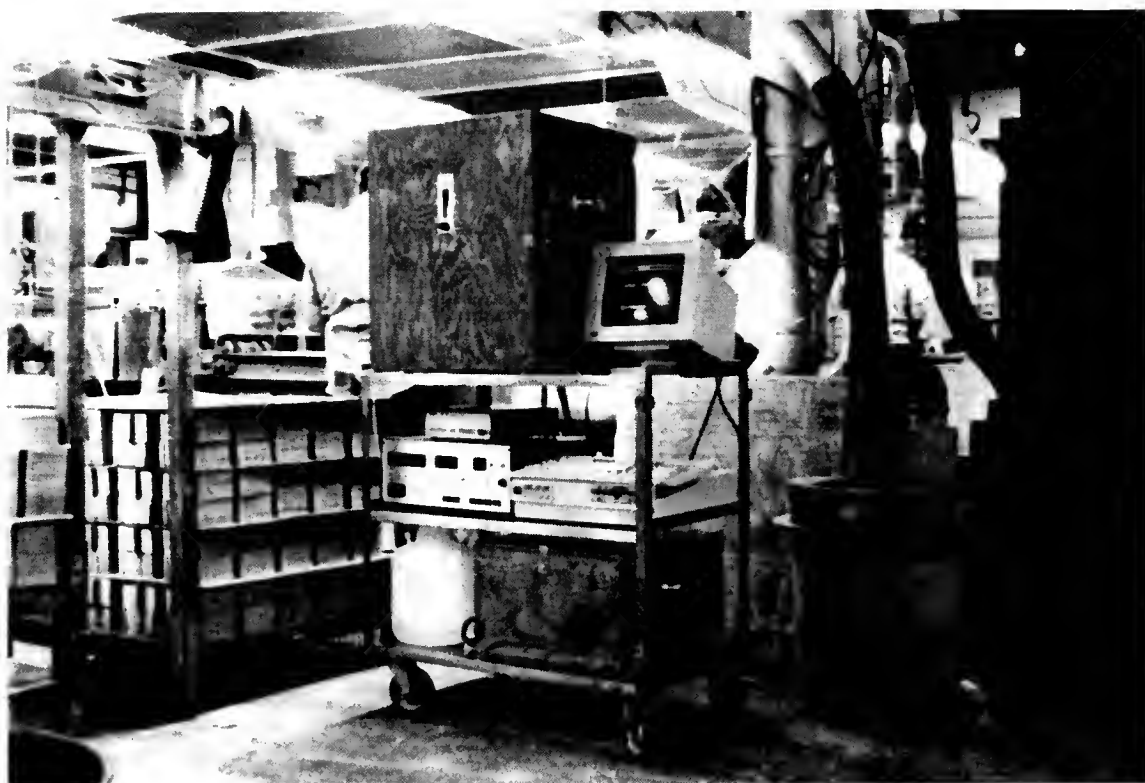


Figure 8-1. Photograph of lead-in-air monitor during tests conducted at plate offbearing process.



Figure 8-2. Photograph of Module 1 inlet section showing position of collocated filter holder relative to Module 1 inlet nozzle.

For the duct sampling tests, the total filter flowrate was adjusted to achieve isokinetic sampling through a 0.42 inch diameter inlet nozzle.

During the week of field tests, fourteen separate sampling runs with collocated filter sampling was performed in the region of the plate off-bearing. To determine the unattended operation capabilities of the monitor, ten unattended sampling runs were performed in this area. Four collocated static sampling runs were also performed in the area of the paste drying oven along with nine unattended tests in this area. Lastly, three collocated sampling runs were performed inside a 10 inch diameter exhaust duct at a plate stacking site.

After each test series, the sampling equipment was returned to the battery plant laboratory. To quantify aerosol transport losses, the Module 1 stainless steel inlet nozzle and teflon inlet tube were removed and their inner surfaces thoroughly rinsed with approximately 100 ml of 0.5M nitric acid with a 200 ppb internal Cu standard. Prior to their reuse, all rinsed components were dried in an oven at 250 °F. The rinses were transferred to separate 150 ml capacity screw-capped polypropylene containers which had been previously cleaned with nitric acid. The particle losses to the total filter nozzle inlet were quantified in a similar manner. All rinse containers were labeled, placed in a lock-tight polypropylene container, and stored at 4 °C until the time of their analysis. Collection filters were placed in separate 150 ml capacity wide-mouth polypropylene containers previously washed with nitric acid. The containers

were labeled and later served as preparation containers during lead extraction in the laboratory.

Following the week of field testing, all equipment was returned to the laboratory. The outside surfaces of all rinse and filter containers were wiped with a 0.5M nitric acid-saturated cloth before the containers were opened. The collected filter samples were extracted in the following manner. Each filter container received 10 ml of 2.5M nitric acid, followed by a 15 minute water bath sonication, followed by a 40 ml addition of D.I. water containing 250 ppm copper standard. The composition of this resulting solution was thus 0.5M nitric acid with a 200 ppb internal copper standard. This solution received an additional 15 minute water bath sonication prior to analysis.

The collected nozzle rinse and extracted filter solutions were analyzed for their lead content using both polarography and GFAA. Three separate analysis were performed for each sample using each analytical technique. Laboratory standards, field standards, and field control samples were also analyzed at this time.

Experimental Results

Prior to analyzing the collected field samples, the 0 to 300 ppb laboratory and field standards were analyzed using both GFAA and polarography. For each technique, three separate analysis were performed for each sample. Tables 8-1 and 8-2 present measured results of the laboratory standards and

Table 8-1
Analysis of Laboratory Lead Standards

<u>Lead Conc. (ppb)</u>	<u>Run No.</u>	<u>Polarography</u>	<u>GFAA</u>
0	1	9	0
	2	7	1
	3	<u>6</u>	<u>0</u>
	Avg	7	0
100	1	99	110
	2	100	106
	3	<u>99</u>	<u>109</u>
	Avg	99	108
200	1	194	218
	2	195	212
	3	<u>195</u>	<u>220</u>
	Avg	195	217
300	1	298	333
	2	295	321
	3	<u>296</u>	<u>327</u>
	Avg	296	327

Table 8-2
Analysis of Field Control Lead Standards

<u>Lead Conc. (ppb)</u>	<u>Run No.</u>	<u>Polarography</u>	<u>GFAA</u>
0	1	11	0
	2	12	0
	3	<u>9</u>	<u>-1</u>
	Avg	11	0
100	1	100	111
	2	99	108
	3	<u>100</u>	<u>109</u>
	Avg	100	109
200	1	192	226
	2	195	218
	3	<u>194</u>	<u>226</u>
	Avg	194	223
300	1	292	336
	2	291	324
	3	<u>293</u>	<u>324</u>
	Avg	292	328

field control standards, respectively. As shown in the tables, the two techniques generally agreed with each other within approximately 10%. The polarography measurements, however, were generally more accurate and displayed greater repeatability than the GFAA analysis. The similarity between the laboratory standards and the field standards indicates that the field containers did not experience significant contamination during the week of field testing.

Collected field filter blanks and nozzle blanks were also analyzed at this time. Results showed that the background lead content of the Module 1 stainless steel inlet, the impactor teflon inlet tube, and the 47 mm filter holder inlet were near the detection limit of the two techniques.

Table 8-3 presents results of the fourteen collocated tests conducted at the plate offbearing process. Presented are the lead concentrations reported by the lead-in-air monitor versus that concentration calculated from the measured filter lead mass and the sampled air volume. Results showed that the monitor's reported concentration agreed well with the total filter measurement. In the measured range of 8 to 30 micrograms/m³, the two values never differed by more than 13%. All measured values were within the occupational permissible exposure limit of 50 micrograms/m³. For the fourteen tests, the average accuracy ratio was 95% with a relative standard deviation of 8.2%. The measured lead mass associated with the inlet rinse represented 4% of the total aerosol sampled. It thus appears that the lead-in-air monitor and the total filter sampler aspirated aerosols of similar mass concentrations.

Table 8-3
Results of Collocated Sampling Performed at Plate Offbearing Operation

<u>Run No.</u>	Lead Conc. (micrograms/m ³)		<u>Ratio</u>
	<u>Lead-in-Air Monitor</u>	<u>Total Filter Sample</u>	
1	26	28	0.93
2	26	30	0.87
3	21	23	0.91
4	21	22	0.95
5	21	22	0.95
6	9	8	1.13
7	14	15	0.93
8	20	23	0.87
9	19	21	0.90
10	16	16	1.00
11	12	11	1.09
12	13	13	1.00
13	6	7	0.94
14	7	8	0.88

An additional ten sampling runs were performed in the area of the plate offbearing. For these tests, the lead-in-air monitor was setup in unattended operating mode and run for five hours without input from the operator. No operational problems were experienced during these tests. Results of these tests are shown in Table 8-4.

Tests were then conducted in the area of the paste drying oven. In this area, three separate collocated filter tests were conducted. Results presented in Table 8-5 again demonstrate the accuracy of the lead-in-air monitor. Note that the monitor was capable of detecting the 78 micrograms/m³ excursion which followed a cleaning cycle of the plate pasting equipment. Following these tests, nine additional tests were performed in this area with the monitor operating in unattended mode. Results of these tests are presented in Table 8-6. Following the ninth test, the monitor correctly detected that the electrolyte supply was insufficient for continued operation and notified the operator of this condition.

The final test series was conducted in the region of the plate stacking operation. In this operation, paper separators and pasted plates are manually loaded into a stacking machine which automatically places the separators between successive plates. Sampling was performed in a 10 inch diameter circular duct which exhausts aerosols generated from the process. A sampling port was installed in a 90° bend located approximately six feet downstream of the process. The stack gas velocity pressure, static pressure, and temperature

Table 8-4
Results of Unattended Sampling Performed at Plate Offbearing Operation

<u>Run No.</u>	<u>Lead Conc.</u> <u>micrograms/m³</u>
1	17
2	16
3	17
4	11
5	16
6	9
7	18
8	11
9	25
10	22

Table 8-5
Results of Collocated Sampling Performed at Paste Drying Operation

<u>Run No.</u>	Lead Conc. (micrograms/m ³)		<u>Ratio</u>
	<u>Lead-in-Air Monitor</u>	<u>Total Filter Sample</u>	
1	76	78	0.97
2	24	26	0.92
3	18	19	0.95

Table 8-6
Results of Unattended Sampling Performed at Paste Drying Operation

<u>Run No.</u>	<u>Lead Conc. micrograms/m³</u>
1	24
2	17
3	23
4	19
5	24
6	21
7	16
8	22
9	22

were measured to be 0.43 inches of water, 29.91 inches of mercury, and 89 °F, respectively.

Three collocated filter tests were performed in this area. For the lead-in-air monitor, a 0.185 inch diameter stainless steel nozzle was used in conjunction with a 39 inch long teflon-lined sampling probe of 0.88 inch inside diameter. For the comparative filter tests, a 0.242 inch diameter stainless steel nozzle was used in conjunction with an in-stack stainless filter holder. Both nozzles were located approximately at the duct centroid and were oriented directly into the airstream. Each of the three tests were conducted for a fifteen minute time period. At the end of the test series, the nozzles and teflon probes were carefully rinsed for their lead content.

As shown in Table 8-7, a significant difference was observed between the monitor's results and those calculated from the filter's lead content. Upon first inspection, it was believed that nozzle and probe losses would explain the lead-in-air monitor's observed underestimation of the lead concentration. However, analysis of the monitor's nozzle, probe, and inlet section lead deposits following this test series showed that only 11% of the sampled aerosol was lost to these surfaces. Probe losses accounted for 7% of the total losses.

It thus appears that the two samplers aspirated significantly different aerosols during the sampling tests. This difference could be possibly explained by the fact that the nozzles were not exactly collocated but were separated by approximately 3 inches. Since the point of sampling was close to the source of

Table 8-7
Results of Collocated Sampling Performed at Plate Stacking Operation

<u>Run No.</u>	Lead Conc. (micrograms/m ³)		<u>Ratio</u>
	<u>Lead-in-Air Monitor</u>	<u>Total Filter Sample</u>	
1	349	697	0.50
2	1084	2831	0.38
3	1323	5545	0.24

aerosol generation, the aerosol may not have been well mixed at the point of sampling. The noted concentration differences may also have resulted due to inadvertent anisokinetic sampling. Considering the large size of the particles produced at this process, anisokinetic sampling could be responsible for a significant fraction of the observed discrepancy. The total filter measurements, therefore, do not accurately gauge the performance of the lead-in-air monitor during these tests. In the absence of an accurate comparison measurement, the total lead mass balance within the lead-in-air monitor's sampling system must be relied upon. For this limited test series, results showed that monitor quantified 89% of the sampled aerosol. Transport losses in the nozzle, probe, and impactor inlet tube accounted for the remaining 11% of the sampled aerosol.

CHAPTER 9

SUMMARY AND RECOMMENDATIONS

A prototype sampling and analysis system has been developed for quantifying lead-based aerosols in the lead-acid battery industry. Design criteria for the instrument were based on measured size distributions and chemical properties specific to aerosols generated during lead-acid battery production. Separate prototype subsystems of air sampling, particle collection, liquid handling, and lead analysis were designed and have been described in detail. Laboratory evaluation of these subsystems verified that they perform essentially as designed.

Field evaluation of the lead-in-air monitor in a battery plant verified that the overall design performed well under conditions of its intended use. For the seventeen collocated static sampling tests performed at two process locations, the mean accuracy of the monitor was measured to be 95% with a relative standard deviation of 7%. Analysis of aerosol losses within the sampling nozzle and impactor inlet tube accounted for the slight deviation from ideal performance. These tests verify that the instrument's particle collection section and lead analysis section perform as designed.

A total of 19 separate static sampling tests were performed at two process locations with the monitor operated in unattended mode. During these

tests, the unit's software provided for automated sequencing of the sample and analysis cycles without input from the operator. Test results were automatically stored on the computer's hard drive for later retrieval and inspection. Mass balance studies of particle transport losses verified that only 4% to 6% of the aspirated aerosol is not quantified due to transport losses. No operational problems were experienced during these unattended tests of the prototype monitor.

A limited series of tests was performed at a plate stacking operation to evaluate the monitor's ability to quantify lead-based particle concentrations in flowing airstreams. Collocated filter sampling indicated that ducted aerosols near the point of their generation may not be uniform across the duct's cross-section. Anisokinetic sampling of large particles in high velocity airstreams can also result in significant measurement error. Due to expected particle bounce and reentrainment within the transport system, actual particle losses were significantly lower than those predicted from worst-case theoretical considerations. As described, recommended modifications to the sampling system may further reduce transport losses with an accompanying increase in the overall accuracy of the monitor's reported results.

The prototype monitor was constructed to verify the overall design approach of an instrument capable of quantifying lead-based particles in the lead-acid battery industry. Laboratory and field evaluation of the prototype confirmed the validity of this approach. A number of recommendations can be

made, however, to improve the instrument's overall performance and reliability. These recommendations should be considered during any future phase of its development.

The primary intended application of the monitor is to serve as an unattended on-line monitor of recirculation air quality. As such, the primary limitation of the prototype instrument is its limited supply of nitric acid electrolyte and nitrogen purge gas. In the current design, the electrolyte and nitrogen supply must be replenished after every 30 and 200 separate analysis runs, respectively. Providing larger available supplies of these two materials would significantly extend the long-term capabilities of the instrument thus requiring less periodic attention from the operator.

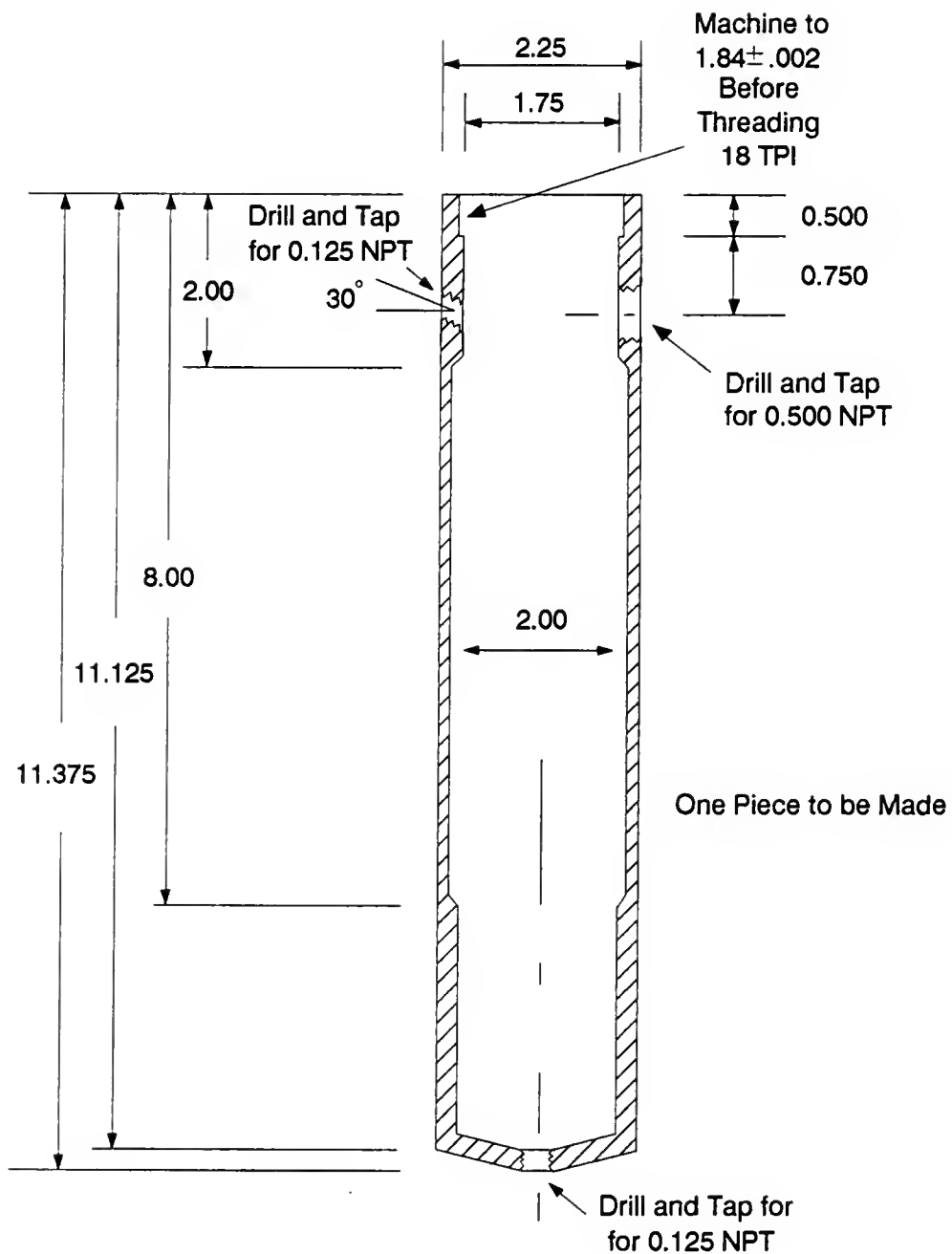
Due to intermittent problems experienced with the liquid handling pumps, it is recommended that they be replaced with pumps of greater reliability and precision. Digitally controlled precision peristaltic pumps should be considered as possible alternatives. Use of extended-life noreprene tubing would minimize the frequency with which the pump's tubing need be replaced.

Future development of the instrument could be devoted to reducing its overall size. If the monitor is intended for static or fixed flowrate sampling, the air sampling system could be reduced in size and simplified. Lower flow requirements for static sampling would directly translate to lower pumping capacities. Perhaps the most significant design simplification could be achieved by developing custom microprocessor-based control circuitry. The use of

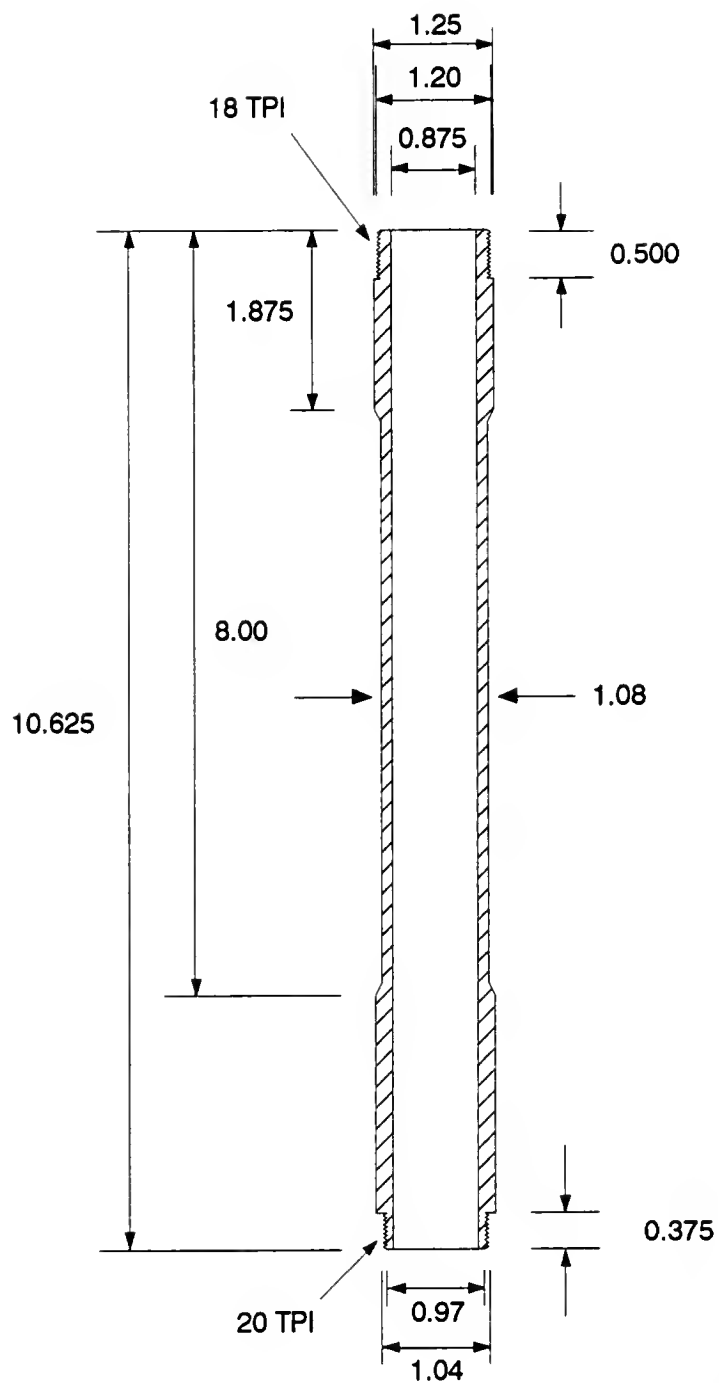
dedicated control circuitry would eliminate the need for the polarograph's control unit, the data acquisition and control system, and the full-sized computer with its accompanying display monitor. This modification alone would greatly improve the portability and reliability of the overall instrument design.

A complete operating manual for the prototype lead-in-air monitor is presented in Appendix E. Included are instructions for setting up the instrument, procedures for performing initial system checks, and instructions for instrument operation during both still air and ducted air field sampling. Instrument maintenance and troubleshooting sections are included in the manual.

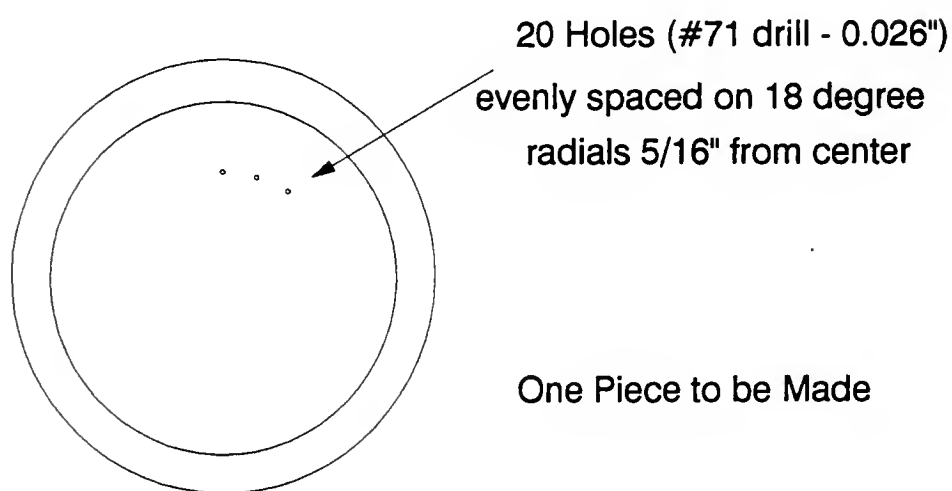
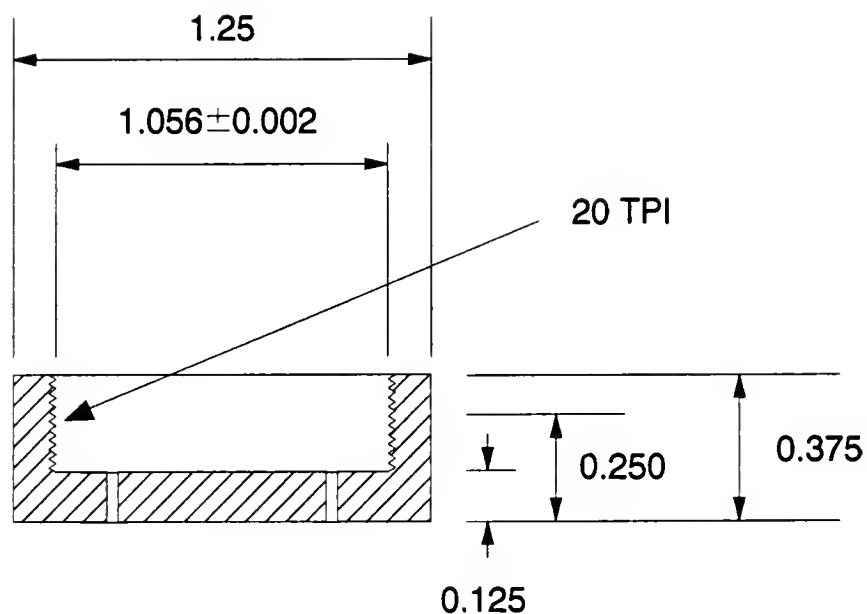
APPENDIX A
DETAIL DRAWINGS OF TEFLON IMPACTOR



Title	Part No.	Material
Housing Body	DRW-5	Teflon
Date	Scale	
August 30, 1991	1 inch = 2 inches	
R.W. Vanderpool	Dimensions are in inches. Tolerances plus or minus 0.01 inches unless otherwise noted	



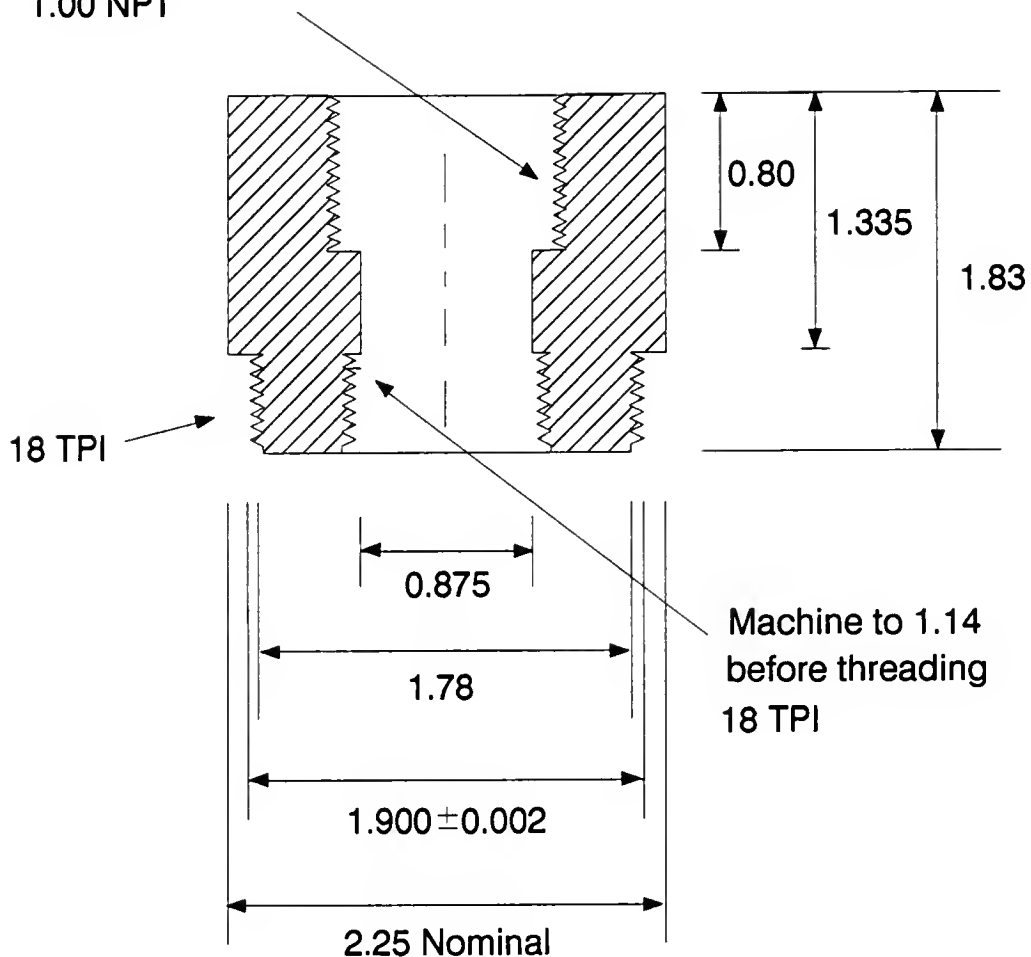
Title	Part No.	Material
Central Tube	DRW-4	Teflon
Date	Scale	
August 30, 1991	1 inch = 2 inches	
R.W. Vanderpool	Dimensions are in inches. Tolerances plus or minus 0.01 inches unless otherwise noted	



One Piece to be Made

Title Impactor Stage	Drawing No. DRW-3	Material Teflon
Date August 30, 1991	Scale 1 inch = 2 inches	
R.W. Vanderpool	Dimensions are in inches. Tolerances plus or minus 0.01 inches unless otherwise noted	

Drill and Tap for
1.00 NPT



One Piece to be Made

Title Housing Inlet	Drawing No. DRW-2	Material Teflon
Date August 30, 1991	Scale Approximate	
R.W. Vanderpool	Dimensions are in inches. Tolerances plus or minus 0.01 inches unless otherwise noted	

APPENDIX B
RESULTS OF IMPACTOR COLLECTION EFFICIENCY TESTS

Table B-1
Results of Impactor Collection Efficiency Tests

Aerodynamic Particle Diameter <u>micrometers</u>	Collection Efficiency (%)		
	<u>10 lpm</u>	<u>20 lpm</u>	<u>30 lpm</u>
0.05	91	87	78
0.10	57	78	82
0.20	27	30	31
0.36	22	32	36
0.50	30	47	54
0.70	30	47	54
1.00	66	78	82
1.40	82	87	90
2.0	95	95	97
3.2	98	99	99
6.0	99.8	99.9	99.9
10.0	99.9	99.9	99.9
20.0	99.9	99.9	99.9

APPENDIX C
CONFIGURATION OF KEITHLEY 575 CHANNELS

Table C-1
Configuration of Keithley 575 Channels

<u>575</u> <u>Connection</u>	<u>Channel No.</u>	<u>Name</u>	<u>Color</u>	<u>Connects to:</u>	<u>Function</u>
J1 (Analog Out)	Ch0	ANOUT0	YEL	DD-7	6 VDC to Decade Board
	Ch1	ANOUT1	BRN	EE-3	0-5 VDC Command Signal to MFC
J12	Ch0	DIGOUT0	RED	DD-1	Powers Fluid Level Switch
	Ch1	DIGOUT1	BLU	DD-5	Activates Nitrogen Solenoid
	Ch2	DIGOUT2	GRY	DD-6	Activates LP1 for Impactor Fill
	Ch3	DIGOUT3	PUR	DD-7	Activates LP2 for Impactor Empty
	Ch4	DIGOUT4	WHT	DD-8	Activates LP3 for Cell Empty
	Ch5	DIGOUT5	PNK	DD-10	Initiates Nitrogen Purge
	Ch6	DIGOUT6	ORG	DD-11	Initiates Stir Function
	Ch7	DIGOUT7	BLK	DD-12	Initiates Drop Dislodge/Dispense
163	Ch8	DIGOUT8	YEL	EE-1	On/Off Control for Pump/Fan

Table C-1 -- continued

<u>575</u> <u>Connection</u>	<u>Channel No.</u>	<u>Name</u>	<u>Color</u>	<u>Connects to:</u>	<u>Function</u>
J1 (Digital Out)	Ch0	ANIN0	RED	EE-5	Reads MFC Output "High"
			BLK	EE-4	Reads MFC Output "Low"
	Ch1	ANIN1	PUR	CC-1	Reads I in I vs. V Scan "High"
			BLU	CC-2	Reads I in I vs. V Scan "Low"
	Ch2	ANIN2	RED	DD-2	Reads Fluid Level Switch "High"
			GRN	GND	Reads Fluid Level Switch "Low"
	Ch3	ANIN3	GRY	CC-4	Reads V in I vs. V Scan "High"
			GRN	GND	Reads V in I vs. V Scan "Low"
	Ch4	ANIN4	RED	CC-3	Checks Status of Scan "High"
			GRN	GND	Checks Status of Scan "Low"

APPENDIX D
PROGRAM LISTING

```

10 '
20 ' SET VALUE FOR STANDARD PRESSURE (IN HG)
30 PRESSURE=29.92
40 ' SET VALUE FOR COPPER STANDARD (PPB)
50 COPPERSTD= 200
60 ' SET TIME FOR AIR SAMPLING (SEC)
70 SAMPLETIME=900
80 ' SET NITROGEN PURGE TIME (SEC)
90 PURGETIME=180
100 ' SET PITOT TUBE CORRECTION FACTOR FOR STANDARD PITOT TUBE
110 CP=.99
120 ' CHANGE FROM CURRENT DIRECTORY TO "RESULTS" DIRECTORY
130 CHDIR "\":CHDIR "\K500\RESULTS"
140 ' DIMENSION BASIC ARRAYS
150 DIM XARRAY(800):DIM YARRAY(800):DIM QARRAY(200):DIM BOX%(2)
160 '
170 '
180 CLS
190 ' INITIALIZE AND CALIBRATE DATA ACQUISITION AND CONTROL SYSTEM
200 CALL KDINIT
210 ' INITIALIZE MODEL 303A PURGE, STIR, AND DISPENSE FUNCTIONS TO
    "OFF"
220 VL!(0)=6!
230 CALL FGWRITE'("anout0",vl!(),"c.volts","nt")
240 VL%(0)=1
250 CALL FGWRITE'("digout5",vl%(),"c.raw.int","nt")
260 CALL FGWRITE'("digout6",vl%(),"c.raw.int","nt")
270 CALL FGWRITE'("digout7",vl%(),"c.raw.int","nt")
280 ' "POWER" LEVEL SWITCH USING DIGITAL HIGH SIGNAL
290 VL%(0)=0
300 CALL FGWRITE'("DIGOUT2",VL%(),"C.RAW.INT","NT")
310 ' INITIALIZE ARRAYS FOR CONTROLLING LIQUID PUMPS LP1, LP2, AND
    LP3
320 BOX%(1)=1:BOX%(2)=0
330 CALL ARMAKE'("LPARRAY",2.,"DIGOUT2")
340 CALL ARPUT'("LPARRAY",1.,2.,"DIGOUT2",1,BOX%(),"C.RAW.INT")
350 CALL ARPUT'("LPARRAY",1.,2.,"DIGOUT3",1,BOX%(),"C.RAW.INT")
360 CALL ARPUT'("LPARRAY",1.,2.,"DIGOUT4",1,BOX%(),"C.RAW.INT")
370 ' DISABLE NON-FATAL ERROR MESSAGES
380 CALL KDWARN'("WARNOFF")
390 '
400 '
410 ' MAIN MENU
420 CLS:CLOSE:SCAN=0:TIMER OFF:LOCATE 1,1:FOR J=1 TO 10:PRINT

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*****";:NEXT J
430 TITLE$="LEAD-IN-AIR MONITOR"
440 LOCATE 2,1:PRINT TAB(40-LEN(TITLE$)/2)TITLE$
450 LOCATE 3,1:FOR J=1 TO 10:PRINT "*****";:NEXT J
460 TITLE$="MAIN MENU"
470 LOCATE 9,1:PRINT TAB(40-LEN(TITLE$)/2)TITLE$
480 TITLE$="-----"
490 LOCATE 10,1:PRINT TAB(40-LEN(TITLE$)/2)TITLE$
500 LOCATE 12,1
510 PRINT TAB(25)"(1)  PERFORM SINGLE SAMPLING AND ANALYSIS TEST"
520 PRINT TAB(25)"(2)  UNATTENDED SAMPLING AND ANALYSIS"
530 PRINT TAB(25)"(3)  ANALYZE SOLUTION ONLY"
540 PRINT TAB(25)"(4)  VIEW OR PRINT PREVIOUS TEST RESULTS"
550 PRINT TAB(25)"(5)  FLUID HANDLING OPTIONS"
560 PRINT TAB(25)"(6)  EXIT PROGRAM"
570  LOCATE 21,1:PRINT  TAB(9);:INPUT  "ENTER  YOUR
      CHOICE";CHOICE:CHOICE=CINT(CHOICE)
580 ' ELIMINATE INVALID CHOICES
590 IF CHOICE<1 OR CHOICE>6 THEN LOCATE 18,1:PRINT TAB(27)"
      ":GOTO 570
600 ON CHOICE GOTO 620,620,2130,4040,6620,5130
610 ' CHECK ELECTROLYTE LEVEL, RINSE SYSTEM, AND TRANSFER 50 ML
      TO IMPACTOR
620 GOSUB 5140:GOSUB 5290:GOSUB 5800
630 IF CHOICE=1 GOTO 830
640 ' INSTRUCTIONS FOR SETTING INSTRUMENT IN UNATTENDED
      OPERATING MODE
650 CLS
660 TITLE$="UNATTENDED OPERATION"
670  LOCATE 1,1:FOR J=1 TO 10:PRINT "*****";:NEXT J
680 LOCATE 2,1:PRINT TAB(40-LEN(TITLE$)/2)TITLE$:LOCATE 9,1
690 LOCATE 3,1:FOR J=1 TO 10:PRINT "*****";:NEXT J
700 LOCATE 9,1:PRINT "YOU HAVE SELECTED FOR THE INSTRUMENT TO
      SAMPLE IN UNATTENDED MODE WITHOUT"
710 PRINT "INPUT FROM THE OPERATOR."
720 PRINT
730 PRINT "THE INSTRUMENT WILL SAMPLE FOR 15 MINUTES AT 30 MINUTE
      INTERVALS."
740 PRINT "THE TEST RESULTS WILL BE AUTOMATICALLY SAVED TO DISK
      FOR LATER INSPECTION."
750 PRINT
760 PRINT "SAMPLING WILL CONTINUE UNTIL MANUALLY ABORTED OR UNTIL
      THE ELECTROLYTE"
770 PRINT "SUPPLY IS INSUFFICIENT FOR CONTINUED OPERATION."

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780 LOCATE 23,23
790 PRINT "PRESS ANY KEY TO CONTINUE ....."
800 IF INKEY$="" GOTO 800
810 '
820 ' ENTER PERTINENT STACK CONDITIONS
830 CLS
840 TITLE$="STACK CONDITIONS"
850 LOCATE 1,1:FOR J=1 TO 10:PRINT "*****";:NEXT J
860 LOCATE 2,1:PRINT TAB(40-LEN(TITLE$)/2)TITLE$
870 LOCATE 3,1:FOR J=1 TO 10:PRINT "*****";:NEXT J
880 LOCATE 6,1:PRINT "PRIOR TO SAMPLING, YOU MUST ENTER THE
      MEASURED STACK CONDITIONS AT THE POINT OF SAMPLING"
890 LOCATE 9,1
900 INPUT "STACK GAS TEMPERATURE (DEG F)";TF
910 PRINT
920 INPUT "STACK GAS VELOCITY PRESSURE (IN H2O). ENTER 0 FOR STATIC
      SAMPLING ";VP
930 IF VP=0 THEN QNOZZLE=15:GOTO 1040
940 ' CALCULATE ACTUAL STACK GAS VELOCITY
950 VEL=85.49*CP*SQR(VP)*SQR((TF+460)/(PRESSURE*29))
960 ' CALCULATE IDEAL NOZZLE SIZE
970 NOZZLE=SQR(4*15000/(3.14*VEL*60*(30.48^3)))*12
980 PRINT:PRINT "FOR THESE CONDITIONS, SELECT A NOZZLE DIAMETER
      CLOSE TO ";USING ".###";NOZZLE;:PRINT " INCHES"
990 LOCATE 15,1
1000 INPUT "ENTER ACTUAL DIAMETER OF AVAILABLE NOZZLE ";DN
1010 ' CALCULATE ACTUAL NOZZLE FLOWRATE REQUIRED TO ACHIEVE
      ISOKINETIC SAMPLING
1020 QNOZZLE=VEL*3.1416*(DN/12)^2*60*28.3/4
1030 ' CONVERT ACTUAL FLOWRATE TO FLOWRATE REQUIRED BY MASS
      FLOW CONTROLLER
1040 QMFC=QNOZZLE*(32+460)/(TF+460)*PRESSURE/29.92
1050 IF VP=0 THEN GOTO 1160
1060 ' CHECK THAT FLOWRATE IS WITHIN 8 TO 30 SLPM
1070 IF QMFC<30 AND QMFC>8 THEN GOTO 1160
1080 IF QMFC<8 THEN LOCATE 17,1:PRINT:PRINT "THE REQUIRED FLOWRATE
      FOR THIS NOZZLE IS TOO LOW. USE A LARGER NOZZLE":BEEP
1090 IF QMFC>30 THEN LOCATE 17,1:PRINT:PRINT "THE REQUIRED
      FLOWRATE FOR THIS NOZZLE IS TOO HIGH. USE A SMALLER
      NOZZLE":BEEP
1100 PRINT:PRINT:PRINT:PRINT TAB(18)"PRESS ANY KEY TO CONTINUE ....."
1110 IF INKEY$="" GOTO 1110
1120 FOR J=15 TO 22
1130 LOCATE J,1:PRINT "

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"
1140 NEXT J
1150 LOCATE 15,1:GOTO 1000
1160 PRINT:PRINT "THE REQUIRED ACTUAL FLOWRATE FOR THIS NOZZLE IS
";PRINT USING "##.##";QNOZZLE;PRINT " LPM"
1170 PRINT:PRINT:INPUT "ENTER DESCRIPTION FOR THIS TEST
";DESCRIPTION$
1180 KEY(5) OFF
1190 '
1200 '
1210 ' ROUTINE FOR AIR SAMPLING OF STACK GAS
1220 CLS:TITLE$="INITIALIZE AIR SAMPLING"
1230 LOCATE 1,1:FOR J=1 TO 10:PRINT "*****";NEXT J
1240 LOCATE 2,1:PRINT TAB(40-LEN(TITLE$)/2)TITLE$
1250 LOCATE 3,1:FOR J=1 TO 10:PRINT "*****";NEXT J
1260 LOCATE 10,5:PRINT "PRIOR TO SAMPLING, MAKE SURE PROBE IS
PROPERLY POSITIONED AND ALIGNED."
1270 LOCATE 18,18:PRINT "PRESS SPACEBAR TO BEGIN AIR SAMPLING ....."
1280 IF INKEY$<>CHR$(32) GOTO 1280
1290 ' READ STARTING TIME FROM COMPUTER
1300 STARTTIME$=LEFT$(TIME$,5)
1310 ' SET COMMAND VOLTAGE TO CONTROL MASS FLOW CONTROLLER.
A ONE VOLT COMMAND SIGNAL IS EQUIVALENT TO 10 LPM AT 32 F.
1320 VL!(0)=QMFC/10
1330 CALL FGWRITE("ANOUT1",VL!(),"C.VOLTS","NT")
1340 ' TURN ON AIR PUMP AND COOLING FAN
1350 VL%(0)=1
1360 CALL FGWRITE("DIGOUT8",VL%(),"C.RAW.INT","NT")
1370 ' LET REMAIN EQUAL THE SPECIFIED SAMPLING TIME (SEC)
1380 REMAIN=SAMPLETIME:TIMER ON
1390 CLS:TITLE$="AIR SAMPLING"
1400 LOCATE 1,1:FOR J=1 TO 10:PRINT "*****";NEXT J
1410 LOCATE 2,1:PRINT TAB(40-LEN(TITLE$)/2)TITLE$
1420 LOCATE 3,1:FOR J=1 TO 10:PRINT "*****";NEXT J
1430 ' SAMPLING WILL CONTINUE UNTIL ELAPSED TIME = SET TIME OR
UNTIL MANUALLY ABORTED BY THE OPERATOR
1440 ' ROUTINE IN EVENT OF MANUAL ABORT
1450 ON KEY(5) GOSUB 1610:KEY(5) ON
1460 LOCATE 20,18:PRINT "PRESS F5 TO TERMINATE SAMPLING ....."
1470 LOCATE 10,20:PRINT "TIME REMAINING =";
1480 ' MEASURE AND RECORD SAMPLING FLOWRATE AT 10 SECOND
INTERVALS
1490 CALL BGREAD("qarray",800.,"anin0",1,"none",1,"nt","TASK10")
1500 CALL INTON'(10,"SEC")

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1510 CALL BGGO'("NT","")
1520 ' ROUTINE FOR RESETTING ELAPSED TIME EVERY 1 SECOND
1530 ON TIMER (1) GOSUB 1560
1540 ' CHECK IF ELAPSED TIME EQUALS THE DESIRED SAMPLING TIME
1550 IF REMAIN>0 THEN GOTO 1550 ELSE 1770
1560 REMAIN=REMAIN-1
1570 MIN=INT(REMAIN/60):SEC=CINT(REMAIN)-MIN*60
1580 LOCATE 10,38:PRINT MIN;"MINUTES ";SEC;"SECONDS "
1590 ELAPSED=TIME-REMAIN
1600 RETURN
1610 CLS:TIMER OFF:TITLE$="SAMPLING ABORTED"
1620 VL%(0)=0
1630 CALL FGWRITE'("DIGOUT8",VL%(),"C.RAW.INT","NT")
1640 RUNTIME=ELAPSED:RUNTIME$=STR$(RUNTIME)
1650 LOCATE 1,1:FOR J=1 TO 10:PRINT "*****";NEXT J
1660 LOCATE 2,1:PRINT TAB(40-LEN(TITLE$)/2)TITLE$
1670 LOCATE 3,1:FOR J=1 TO 10:PRINT "*****";NEXT J
1 6 8 0 E L A P S E D
    =TIME-REMAIN:MIN=INT(ELAPSED/60):SEC=CINT(ELAPSED)-MIN*60
1690 LOCATE 7,1:PRINT "THE ELAPSED SAMPLING TIME WAS ";MIN;"MINUTES
    ";SEC;"SECONDS"
1700 ' CHECK THAT SAMPLING TIME WAS AT LEAST 5 MINUTES. IF NOT
    ABORT THE RUN
1710 GOTO 1770
1720 IF ELAPSED<300 THEN LOCATE 10,1:PRINT "THIS SAMPLING TIME WAS
    INSUFFICIENT FOR ACCURATE TEST RESULTS"
1730 LOCATE 12,1:PRINT "NO LEAD ANALYSIS OF THE COLLECTED SAMPLE
    WILL BE PERFORMED."
1740 IF ELAPSED <300 THEN LOCATE 20,5:PRINT "PLEASE WAIT FOR
    INSTRUMENT TO RINSE ....."
1750 IF ELAPSED<300 THEN GOTO 420
1760 ' TURN OFF AIR PUMP AND COOLING FAN
1770 ELAPSED=SAMPLETIME:TIMER OFF:VL%(0)=0
1780 CALL FGWRITE'("DIGOUT8",VL%(),"C.RAW.INT","NT")
1790 CALL INTOFF
1800 CALL BGCLEAR
1810 LP!=0
1820 ' FIND NUMBER OF POINTS STORED IN FLOW ARRAY
1830 CALL ARLASTP'("QARRAY",LP!)
1840 LP=LP!:QAVG=0:SUM=0
1850 ' TRANSFER CONTENTS OF KDAC ARRAY TO GWBASIC ARRAY
1860 FOR N=1 TO LP
1870 CALL ARGET'("QARRAY",N!,N!,"ANINO",1,QARRAY!(),"C.VOLTS")
1880 QARRAY(N)=QARRAY!(0)

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1890 NEXT N
1900 ' DISCARD FLOW MEASUREMENT VALUE AT TIME ZERO. CALCULATE
      AVERAGE FLOWRATE USING REMAINING VALUES.
1910 FOR N=2 TO LP
1920 SUM=SUM + 10*QARRAY(N)
1930 NEXT N
1940 QAVG=SUM/(LP-1)
1950 ' CALCULATE FLOWRATE BASED ON OCCUPATIONAL STANDARD
      TEMPERATURE OF 77 F
1960 QSTD=QAVG*(77+460)/(32+460)
1970 AVGFLOWRATE=QSTD
1980 ' CALCULATE THE PERCENT ISOKINETIC FOR THIS RUN
1990 ISO=QAVG/QMFC*100
2000 ' DISPLAY ERROR MESSAGE IF NOT WITHIN 10% ISOKINETIC
2010 IF ISO<90 THEN CLS:LOCATE 10,1:PRINT "THE FLOWRATE FOR THIS
      TEST WAS INSUFFICIENT FOR ISOKINETIC SAMPLING. ROTATE THE AIR
      PUMP RECIRCULATION VALVE CLOCKWISE APPROXIMATELY 1 TURN
      AND REPEAT TEST"
2020 IF ISO>110 THEN CLS:LOCATE 10,1:PRINT "THE FLOWRATE FOR THIS
      RUN WAS TOO HIGH FOR ACCURATE TEST RESULTS. IF THIS
      CONDITION PERSISTS ON SUBSEQUENT RUNS, CHECK THE
      OPERATION OF THE MASS FLOW CONTROLLER."
2030 IF ISO>89 AND ISO<111 THEN GOTO 2060
2040 LOCATE 20,18:PRINT "PRESS ANY KEY TO CONTINUE ....."
2050 IF INKEY$="" THEN GOTO 2050
2060 KEY(5) OFF
2070 ON KEY(5) GOSUB 420
2080 KEY (5) ON
2090 RUNTIME=ELAPSED:RUNTIME$=STR$(RUNTIME)
2100 CLS:LOCATE 10,10:PRINT "TRANSFERRING SOLUTION TO CELL
      FOR ANALYSIS. PLEASE WAIT ....."
2110 ' TRANSFER SOLUTION FROM IMPACTOR TO POLAROGRAPHIC CELL
2120 GOSUB 5930
2130 IF CHOICE=3 THEN QMFC=1:TIME=1
2140 IF CHOICE =3 THEN CLS:PRINT:PRINT:INPUT "ENTER DESCRIPTION FOR
      THIS TEST ";DESCRIPTION$
2150 ' ANALYZE COLLECTED SAMPLE
2160 ' SET THE INITIAL CURRENT RANGE TO 20K RESISTOR (10 MICROAMP
      SCALE)
2170 RANGE=6
2180 REMAIN=PURGETIME
2190 ' BEGIN NITROGEN PURGE OF POLAROGRAPHIC CELL
2200 VL%(0)=1
2210 ' OPEN NITROGEN SOLENOID
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2220 CALL FGWRITE'("DIGOUT1",VL%(),"C.RAW.INT","NT")
2230 VL%(0)=0
2240 ' INITIATE PURGE ON MODEL 303A
2250 CALL FGWRITE'("DIGOUT5",VL%(),"C.RAW.INT","NT")
2260 ' INITIATE STIR ON MODEL 303A
2270 CALL FGWRITE'("DIGOUT6",VL%(),"C.RAW.INT","NT")
2280 TIMER OFF:TIMER ON
2290 CLS:TITLE$="LEAD ANALYSIS"
2300 LOCATE 1,1:FOR J=1 TO 10:PRINT "*****";:NEXT J
2310 LOCATE 2,1:PRINT TAB(40-LEN(TITLE$)/2)TITLE$
2320 LOCATE 3,1:FOR J=1 TO 10:PRINT "*****";:NEXT J
2330 LOCATE 20,17:PRINT "VERIFY ROTAMETER SETTING IS BETWEEN
      0.03 AND 0.05 LPM"
2340 LOCATE 10,20:PRINT "PURGE TIME REMAINING = ";
2350 ' ROUTINE FOR RESETTING AND DISPLAYING ELAPSED TIME EVERY 1
      SECOND
2360 ON TIMER (1) GOSUB 2380
2370 IF REMAIN>0 THEN GOTO 2370 ELSE 2430
2380 REMAIN= REMAIN-1
2390 MIN=INT(REMAIN/60):SEC=CINT(REMAIN)-MIN*60
2400 LOCATE 10,44:PRINT MIN;"MINUTES ";SEC;"SECONDS "
2410 RETURN
2420 ' CLOSE NITROGEN SOLENOID AND DISCONTINUE PURGE AND STIR
      FUNCTIONS ON MODEL 303A
2430 TIMER OFF
2440 VL%(0)=0
2450 CALL FGWRITE'("DIGOUT1",VL%(),"C.RAW.INT","NT")
2460 VL%(0)=1
2470 CALL FGWRITE'("DIGOUT5",VL%(),"C.RAW.INT","NT")
2480 CALL FGWRITE'("DIGOUT6",VL%(),"C.RAW.INT","NT")
2490 '
2500 ' INITIALIZE ARRAYS FOR STORING DATA FROM ANALYSIS OF
      COLLECTED LEAD
2510 ON KEY(5) GOSUB 3920
2520 KEY (5) ON
2530 ' RESET DECADE BOARD RELAYS TO "OPEN" POSITION
2540 FOR J=4 TO 8
2550 VL%(0)=1:J$=STR$(J)
2560 BOARD$="PCM"+RIGHT$(J$,1)
2570 CALL FGWRITE'(BOARD$,VL%(),"C.RAW.INT","NT")
2580 NEXT J
2590 ' SET RANGE OF CURRENT SCAN
2600 VL%(0)=0
2610 RANGE$=STR$(RANGE)

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2620 RANGE$="PCM"+RIGHT$(RANGE$,1)
2630 ' PLACE DECADE RESISTANCE BOARD IN-LINE
2640 CALL FGWRITE'("PCM9",VL%(),"C.RAW.INT","NT")
2650 CALL FGWRITE'(RANGE$,VL%(),"C.RAW.INT","NT")
2660 VL%(0)=0
2670 ' INITIATE "SCAN" ON MODEL 264
2680 CALL FGWRITE'("PCM1",VL%(),"C.RAW.INT","NT")
2690 VL%(0)=1
2700 CALL FGWRITE'("PCM1",VL%(),"C.RAW.INT","NT")
2710 ' INITIALIZE VARIABLE ARRAYS
2720 STAT1%=1
2730 RR=0:LP!=0!
2740 LOCATE 10,20:PRINT "DEPOSITING LEAD ON ELECTRODE ..... "
2750 LOCATE 20,17:PRINT "          PLEASE WAIT ..... "
2760 ' ROUTINE FOR DETECTING WHEN MODEL 264 HAS BEGUN THE
      SCAN.  USE THE AVERAGE OF 10 READINGS
2770 FOR J=1 TO 10
2780 CALL FGREAD'("ANIN4","NONE",VL!(),"C.VOLTS","NT")
2790 SUM=SUM+VL!(0):NEXT J
2800 AVG=SUM/10:IF AVG>2 THEN SUM=0:AVG=0:GOTO 2770
2810 SUM=0:AVG=0
2820 CLS:TITLE$="LEAD ANALYSIS"
2830 LOCATE 1,1:FOR J=1 TO 10:PRINT "*****";:NEXT J
2840 LOCATE 2,1:PRINT TAB(40-LEN(TITLE$)/2)TITLE$
2850 LOCATE 3,1:FOR J=1 TO 10:PRINT "*****";:NEXT J
2860 LOCATE 10,20:PRINT "          SCAN DETECTED ..... "
2870 SCAN=1
2880 '
2890 ' READ OUTPUTS FROM MODEL 264 (CURRENT VS SCAN VOLTAGE)
      AND STORE IN KDAC ARRAYS
2900 CALL BGREAD'("XARRAY",800., "ANIN3",1,"NONE",1,"NT","TASK1")
2910 CALL BGREAD'("YARRAY",800., "ANIN1",1,"NONE",1,"NT","TASK2")
2920 CALL INTON'(200,"MIL")
2930 CALL BGGO'("NT","")
2940 START=TIMER
2950 CALL BGSTATUS'("TASK1",STAT1%)
2960 ' ROUTINE FOR DETECTING WHEN MODEL 264 HAS COMPLETED THE
      SCAN.  USE THE AVERAGE OF 10 READINGS
2970 FOR J=1 TO 10
2980 CALL FGREAD'("ANIN4","NONE",VL!(),"C.VOLTS","NT")
2990 SUM=SUM+VL!(0):NEXT J
3000 AVG=SUM/10:IF AVG>3 THEN LOCATE 10,20:PRINT "SCAN COMPLETED.
      PERFORMING CALCULATIONS ..... ":GOTO 3030
3010 AVG=0:SUM=0:GOTO 2970

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3020 IF (STAT1%)=0! THEN 2950
3030 CALL INTOFF
3040 ' FIND LOCATION OF LAST POINT STORED IN ARRAY
3050 CALL ARLASTP'("YARRAY",LP!)
3060 TIMER OFF:VL%(0)=0
3070 CALL FGWRITE'("DIGOUT1",VL%(),"C.RAW.INT","NT")
3080 LP=LP!
3090 ' TRANSFER CONTENTS FROM KDAC ARRAY TO BASIC ARRAY
3100 FOR N=1 TO LP
3110 CALL ARGET'("XARRAY",N!,N!,"ANIN3",1,XARRAY!(),"C.VOLTS")
3120 XARRAY(N)=XARRAY!(0): IF XARRAY(N) < 0 THEN
      XARRAY(N)=ABS(XARRAY(N)) ELSE XARRAY(N)=-XARRAY(N)
3130 CALL ARGET'("YARRAY",N!,N!,"ANIN1",1,YARRAY!(),"C.VOLTS")
3140 YARRAY(N)=YARRAY!(0):IF ABS(YARRAY(N))<.01 THEN YARRAY(N)=0
3150 NEXT N
3160 ' ROUTINE FOR CHECKING AND DISCARDING SPURIOUS VALUES AND
      FOR DETERMINING PEAKS VALUES OF LEAD AND COPPER. FIRST, SET
      PREVIOUS VALUE = 0
3170 PB=0:CU=0
3180 FOR N=1 TO LP
3190 IF ABS(YARRAY(N))>7 THEN YARRAY(N)=0
3200 IF ABS(YARRAY(N)-YARRAY(N-1))>1 THEN YARRAY(N)=YARRAY(N-1)
3210 IF N<100 OR N>160 THEN 3240
3220 IF ABS(YARRAY(N))<ABS(YARRAY(N-1)) THEN GOTO 3240
3230 IF ABS(YARRAY(N))>ABS(PB) THEN PB=YARRAY(N):XX=N
3240 IF N<250 OR N>400 THEN 3260
3250 IF ABS(YARRAY(N))>ABS(CU) THEN CU=YARRAY(N):YY=N
3260 NEXT N
3270 ' CHECK TO SEE IF CURRENT RANGE WAS EXCEEDED DURING THE
      SCAN. IF SO, INCREASE RANGE AND REPEAT THE SCAN
3280 IF ABS(PB)>4.5 OR ABS(CU)>4.5 THEN RANGE=RANGE+1
3290 IF ABS(PB)>4.5 OR ABS(CU)>4.5 THEN CALL ARDEL'("XARRAY")
3300 IF ABS(PB)>4.5 OR ABS(CU)>4.5 THEN CALL ARDEL'("YARRAY")
3310 IF ABS(PB)>4.5 OR ABS(CU)>4.5 THEN GOTO 2180
3320 IF RANGE=6 AND ABS(CU)<.3 THEN GOTO 6480
3330 ' CALCULATE LEAD CONCENTRATION (PPB) IN SOLUTION
3340 LIQUIDPB = PB/CU*COPPERSTD*2.33
3350 ' CALC LEAD-IN-AIR CONCENTRATION (MICROGRAMS PER CUBIC
      METER)
3360 IF CHOICE=3 THEN AIRPB=0:QSTD=0:ISO=0:GOTO 3380
3370 AIRPB=LIQUIDPB*50/(QSTD*SAMPLETIME)*60
3380 ' DELETE CONTENTS OF BASIC ARRAYS AND REDIMENSION
3390 PRINT "xx=";XX:PRINT
3400 ERASE XARRAY:ERASE YARRAY:DIM XARRAY(800):DIM YARRAY(800)

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3410 '
3420 ' LISTING OF ANALYSIS RESULTS
3430 CLS
3440 TITLE$="SUMMARY OF RESULTS"
3450 LOCATE 1,1:FOR J=1 TO 10:PRINT "*****";:NEXT J
3460 LOCATE 2,1:PRINT TAB(40-LEN(TITLE$)/2)TITLE$
3470 LOCATE 3,1:FOR J=1 TO 10:PRINT "*****";:NEXT J
3480 LOCATE 10,10:PRINT "CALCULATED LIQUID LEAD CONC=
";CINT(LIQUIDPB);" PARTS/BILLION "
3490 LOCATE 11,10:PRINT "CALCULATED AIR LEAD CONC= ";CINT(AIRPB);"
MICROGRAMS PER CUBIC METER"
3500 LOCATE 13,10:PRINT "COPPER PEAK= ";CU;" VOLTS"
3510 LOCATE 14,10:PRINT "LEAD PEAK= ";PB;" VOLTS"
3520 LOCATE 16,10:PRINT "VOLUME SAMPLED (STANDARD LITERS)
";CINT(QSTD*SAMPLETIME/60)
3530 LOCATE 15,10:PRINT "% ISOKINETIC= ";CINT(ISO)
3540 ' IF SCAN HAS TAKEN PLACE, DELETE CONTENTS OF KDAC ARRAYS
3550 IF SCAN=1 THEN CALL ARDEL'("XARRAY")
3560 IF SCAN=1 THEN CALL ARDEL'("YARRAY")
3570 IF CHOICE=3 THEN GOTO 3590
3580 IF SCAN=1 THEN CALL ARDEL'("QARRAY")
3590 ' IF OPERATING IN UNATTENDED MODE, DISPLAY THE TEST RESULTS
FOR 30 SECONDS THEN CONTINUE OPERATION
3600 IF CHOICE=2 THEN GOTO 6910
3610 LOCATE 23,20:PRINT "PRESS ANY KEY TO CONTINUE ....."
3620 IF INKEY$="" THEN GOTO 3620
3630 ' ROUTINE TO SAVE TEST RESULTS TO HARD DRIVE
3640 ' USE CURRENT DATE AS FILENAME
3650 FILENAME$=LEFT$(DATE$,6) + RIGHT$(DATE$,2)
3660 OPEN "R",#1,FILENAME$,120
3670 FIELD#1, 2 AS N$, 5 AS START$, 4 AS ELAPSED$, 4 AS AVGFLOWRATE$,
4 AS LIQUIDPB$, 4 AS AIR$, 30 AS DESC$
3680 ' FIND LOCATION OF LAST RECORD STORED ON THIS DAY
3690 N%=1
3700 GET#1,N%
3710 IF CVI(N$)<>0 THEN N%=N%+1:GOTO 3700
3720 LSET N$=MKI$(N%)
3730 LSET START$=STARTTIME$
3740 IF CHOICE=3 THEN LSET START$="0"
3750 IF CHOICE=3 THEN ELAPSED=0:AVGFLOWRATE=0:AIRPB=0
3760 LSET ELAPSED$=MKI$(ELAPSED)
3770 LSET AVGFLOWRATE$=MKI$(AVGFLOWRATE)
3780 LSET LIQUIDPB$=MKI$(LIQUIDPB)
3790 LSET AIR$=MKI$(AIRPB)

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3800 LSET DESC$=DESCRIPTION$
3810 PUT#1, N%
3820 CLOSE
3830 TIMER OFF
3840 IF CHOICE=3 THEN GOTO 410
3850 ' IF OPERATING IN UNATTENDED MODE, REPEAT MAIN CYCLE
3860 ' EXECUTE MAIN RINSE ROUTINE
3870 GOSUB 5290
3880 IF QQ=1 AND CHOICE=2 THEN 3910
3890 IF CHOICE=2 THEN GOSUB 5800
3900 IF CHOICE=2 THEN GOTO 1300
3910 QQ=0:CLS:GOTO 420
3920 ' ROUTINE FOLLOWING MANUAL ABORT OF SCAN
3930 ' IF SCAN HAS TAKEN PLACE, DELETE CONTENTS OF KDAC ARRAY
3940 IF SCAN=1 THEN CALL ARDEL("XARRAY")
3950 IF SCAN=1 THEN CALL ARDEL("YARRAY")
3960 TIMER OFF
3970 CALL INTOFF
3980 ' ACTIVATE "STOP" OF 264A
3990 VLI(0)=0:CALL FGWRITE("PCM2",VLI(),"C.VOLTS","NT")
4000 VLI(0)=1:CALL FGWRITE("PCM2",VLI(),"C.VOLTS","NT")
4010 GOTO 420
4020 '
4030 ' ROUTINE FOR VIEWING RESULTS OF PREVIOUS TESTS
4040 CLS:TITLE$="VIEW PREVIOUS TEST RESULTS"
4050 ON KEY(5) GOSUB 4050:KEY(5) ON
4060 LOCATE 1,1:FOR J=1 TO 10:PRINT "*****";NEXT J
4070 LOCATE 2,1:PRINT TAB(40-LEN(TITLE$)/2)TITLE$
4080 LOCATE 3,1:FOR J=1 TO 10:PRINT "*****";NEXT J
4090 LOCATE 7,4:PRINT "TEST RESULTS ARE SAVED UNDER FILES NAMES
SPECIFIED BY THE SAMPLING DATE"
4100 TITLE$="MENU OF TEST RESULTS"
4110 LOCATE 12,1:PRINT TAB(40-LEN(TITLE$)/2)TITLE$
4120 TITLE$="-----"
4130 LOCATE 13,1:PRINT TAB(40-LEN(TITLE$)/2)TITLE$
4140 LOCATE 15,1
4150 PRINT TAB(25) "(1) LIST EXISTING FILE NAMES"
4160 PRINT TAB(25) "(2) VIEW ARCHIVED TEST RESULTS"
4170 PRINT TAB(25) "(3) PRINT ARCHIVED TEST RESULTS"
4180 PRINT TAB(25) "(4) EXIT TO MAIN MENU"
4190 LOCATE 20,15:INPUT "ENTER YOUR CHOICE
";CHOICE:CHOICE=CINT(CHOICE)
4200 IF CHOICE<1 OR CHOICE>4 THEN LOCATE 20,15:PRINT "
":GOTO 4190

```

```

4210 ON CHOICE GOTO 4220,4260,4700, 420
4220 CLS:FILES
4230 LOCATE 22,20:PRINT "PRESS ANY KEY TO CONTINUE ....."
4240 IF INKEY$ <> CHR$(32) THEN 4240
4250 GOTO 4040
4260 CLS:TITLE$="VIEW TEST RESULTS"
4270 LOCATE 1,1:FOR J=1 TO 10:PRINT "*****";:NEXT J
4280 LOCATE 2,1:PRINT TAB(40-LEN(TITLE$)/2)TITLE$
4290 LOCATE 22,20:PRINT "PRESS <RETURN> KEY TO RETURN TO THE
      MENU ....."
4300 ' DELETE PREVIOUS FILE NAMES FROM MEMORY
4310 FILE$="":FILENAME$=""
4320 LOCATE 12,1:INPUT "ENTER THE FILE NAME YOU WISH TO VIEW";FILE$
4330 IF LEN(FILE$)<6 THEN 4040
4340 ' CONVERT IMPROPER MM/DD/YY FORMAT TO MM-DD-YY FORMAT
4350 FOR J=1 TO LEN(FILE$)
4360 IF ASC(MID$(FILE$,J,1))=47 THEN FILE$(J)="-" ELSE
      FILE$(J)=MID$(FILE$,J,1)
4370 FILENAME$=FILENAME$+FILE$(J)
4380 NEXT J
4390 ' CONVERT IMPROPER M-DD-YY FORMAT TO MM-DD-YY FORMAT
4400 FOR J=1 TO LEN(FILENAME$)
4410 IF J>1 THEN GOTO 4420 ELSE IF ASC(MID$(FILENAME$,J+1,1))=45
      THEN FILENAME$="0"+FILENAME$
4420 IF J<>4 THEN GOTO 4430 ELSE IF ASC(MID$(FILENAME$,J+1,1))=45
      THEN FILENAME$=LEFT$(FILENAME$,3)+"0"+RIGHT$(FILENAME$,4)
4430 NEXT J
4440 OPEN "R",#1,FILENAME$,120
4450 FIELD#1, 2 AS N$, 5 AS START$, 4 AS ELAPSED$, 4 AS AVGFLOWRATE$,
      4 AS LIQUIDPB$, 4 AS AIR$, 30 AS DESC$
4460 N%=1:GET#1, N%
4470 ' CHECK THAT SPECIFIED FILENAME EXISTS
4480 IF CVI(N$)=0 THEN PRINT:PRINT "FILENAME DOES NOT EXIST. HIT ANY
      KEY ....."
4490 IF CVI(N$)=0 THEN CLOSE:KILL FILENAME$
4500 IF CVI(N$)=0 THEN FILENAME$="": IF INKEY$="" THEN 4500
4510 IF CVI(N$)=0 THEN GOTO 4040
4520 '
4530 ' LOAD AND DISPLAY CONTENTS OF SPECIFIED FILENAME
4540 CLS:COUNTER=0:TITLE$="FILENAME = " + FILENAME$:PRINT
      TAB(40-LEN(TITLE$)/2)TITLE$
4550 LOCATE 5,1
4560 PRINT TAB(15)"RUN";TAB(21)"FLOW";TAB(29)"LIQUID";TAB(39)"AIR"
4570 PRINT TAB( 7)"START";TAB(14)"TIME";TAB(21)"RATE";TAB(28)"PB

```

```

CONC";TAB(38)"PB CONC"
4580 PRINT TAB(2)"RUN#";TAB(8)"TIME";TAB(14)"(SEC)";TAB(21)"(LPM)"
;TAB(29)"(PPB)";TAB(37)"(UG/M^3)";TAB(49)"DESCRIPTION"
4590 FOR J=1 TO 80:PRINT"-";NEXT J:PRINT
4600 PRINT TAB(1) USING "###";CVI(N$);PRINT TAB(7)START$;PRINT
TAB(14) USING "###";CVI(ELAPSED$);PRINT TAB(22) USING
"##";CVI(AVGFLOWRATE$);PRINT TAB(29) USING
"####";CVI(LIQUIDPB$);PRINT TAB(38) USING "####";CVI(AIR$);
PRINT TAB(48)DESC$
4610 N%=N%+1
4620 ' LIMIT DISPLAY TO 10 RECORDS
4630 COUNTER=COUNTER+1:IF COUNTER> 9 THEN GOTO 4650
4640 GET#1,N%:IF CVI(N$)<>0 THEN 4600
4650 LOCATE 23,20:PRINT "PRESS ANY KEY TO CONTINUE ....."
4660 IF INKEY$="" THEN GOTO 4660
4670 ' LIMIT DISPLAY TO 10 RECORDS
4680 GET#1,N%
4690 IF COUNTER> 9 THEN GOTO 4540 ELSE CLOSE:GOTO 4040
4700 ' ROUTINE FOR PRINTING
4710 CLS:TITLE$="PRINT PREVIOUS TEST RESULTS"
4720 LOCATE 1,1:FOR J=1 TO 10:PRINT "*****";NEXT J
4730 LOCATE 2,1:PRINT TAB(40-LEN(TITLE$)/2)TITLE$
4740 LOCATE 3,1:FOR J=1 TO 10:PRINT "*****";NEXT J
4750 LOCATE 7,17:PRINT "A PARALLEL PRINTER MUST BE CONNECTED TO
PORT#1"
4760 ' DELETE PREVIOUS FILE NAMES FROM MEMORY
4770 FILE$="":FILENAME$=""
4780 LOCATE 12,1:INPUT "ENTER THE FILE NAME YOU WISH TO PRINT";FILE$
4790 IF LEN(FILE$)<6 THEN 4040
4800 ' CONVERT IMPROPER DD/MM/YY FORMAT TO DD-MM-YY FORMAT
4810 FOR J=1 TO LEN(FILE$)
4820 IF ASC(MID$(FILE$,J,1))=47 THEN FILE$(J)="-" ELSE
FILE$(J)=MID$(FILE$,J,1)
4830 FILENAME$=FILENAME$+FILE$(J)
4840 NEXT J
4850 ' CONVERT IMPROPER D-M-YY FORMAT TO DD-MM-YY FORMAT
4860 FOR J=1 TO LEN(FILENAME$)
4870 IF J=1 AND ASC(MID$(FILENAME$,J+1,1))=45 THEN
FILENAME$="0"+FILENAME$
4880 IF J=4 AND ASC(MID$(FILENAME$,J+1,1))=45 THEN
FILENAME$=LEFT$(FILENAME$,3)+"0"+RIGHT$(FILENAME$,4)
4890 NEXT J
4900 CLOSE
4910 OPEN "R",#1,FILENAME$,120

```



```

4920 FIELD#1, 2 AS N$, 5 AS START$, 4 AS ELAPSED$, 4 AS AVGFLOWRATES$,
      4 AS LIQUIDPB$, 4 AS AIR$, 30 AS DESC$
4930 N%=1:GET#1, N%
4940 ' CHECK THAT SPECIFIED FILENAME EXISTS
4950 IF CVI(N$)=0 THEN PRINT:PRINT "FILENAME DOES NOT EXIST. HIT ANY
      KEY ....."
4960 IF CVI(N$)=0 THEN CLOSE:KILL FILENAME$
4970 IF CVI(N$)=0 THEN FILENAME$="": IF INKEY$="" THEN 4500
4980 IF CVI(N$)=0 THEN CLOSE: GOTO 4040
4990 LOCATE 20,20:PRINT "PRESS ANY KEY TO BEGIN PRINTING ....."
5000 IF INKEY$="" THEN GOTO 5000
5010 '
5020 ' LOAD AND PRINT CONTENTS OF SPECIFIED FILENAME
5030 TITLE$ = "FILENAME = " + FILENAME$:LPRINT
      TAB(40-LEN(TITLE$)/2)TITLE$:LPRINT:LPRINT
5040 LPRINT TAB(15)"RUN";TAB(21)"FLOW";TAB(29)"LIQUID";TAB(39)"AIR"
5050 LPRINT TAB( 7)"START";TAB(14)"TIME";TAB(21)"RATE";TAB(28)"PB
      CONC";TAB(38)"PB CONC"
5060 LPRINTTAB(2)"RUN#";TAB(8)"TIME";TAB(14)"(SEC)";TAB(21)"(LPM)"
      ;TAB(29)"(PPB)" ;TAB(37)"(UG/M^3)";TAB(49)"DESCRIPTION"
5070 FOR J=1 TO 80:LPRINT"-";:NEXT J:LPRINT
5080 LPRINT TAB(1) USING "###";CVI(N$);:LPRINT TAB(7)START$;: LPRINT
      TAB(14) USING "###";CVI(ELAPSED$);: LPRINT TAB(22) USING
      "##";CVI(AVGFLOWRATES$);:LPRINT TAB(29) USING
      "####";CVI(LIQUIDPB$);:LPRINT TAB( 38) USING "####";CVI(AIR$);:
      LPRINT TAB(48)DESC$
5090 N%=N%+1
5100 GET#1,N%:IF CVI(N$)<>0 THEN 5080
5110 CLOSE:GOTO 4040
5120 ' TERMINATE PROGRAM EXECUTION
5130 CHDIR "\":CHDIR "\K500":CLOSE:END
5140 ' CHECK ELECTROLYTE LEVEL SWITCH POSITION. USE AVERAGE OF
      10 READINGS
5150 VL%(0)=1
5160 CALL FGWRITE("DIGOUT0",VL%(),"C.RAW.INT","NT")
5170 SUM=0:AVG=0
5180 VL!(0)=0
5190 FOR J=1 TO 10
5200 CALL FGREAD("ANIN2","NONE",VL!(),"C.VOLTS","NT")
5210 SUM=SUM+VL!(0):NEXT J
5220 AVG=SUM/10:IF AVG>3 THEN RETURN
5230 CLS:LOCATE 7, 9:PRINT "NOTICE: ELECTROLYTE SUPPLY IS
      INSUFFICIENT FOR CONTINUED OPERATION"
5240 LOCATE 10,24: PRINT "REFILL RESERVOIR BEFORE CONTINUING"

```

```

5250 FOR J=1 TO 5:BEEP:FOR K=1 TO 4000:NEXT K:NEXT J
5260 LOCATE 20,26: PRINT "PRESS ANY KEY TO CONTINUE ....."
5270 IF INKEY$="" THEN GOTO 5270
5280 GOTO 420
5290 ' MAIN RINSE ROUTINE
5300 CLS:LOCATE 10,20:PRINT "CLEANING POLAROGRAPHIC CELL. PLEASE
      WAIT ....."
5310 STAT%=10
5320 ' LP1 PUMP 50 ML (66 CYCLES)
5330 CALL BGWRITE'("LPARRAY","DIGOUT2",1,66,"NT","TASK2")
5340 ' TURN ON STIRRING OF POLAROGRAPHIC CELL DURING RINSE
5350 VL%(0)=0
5360 CALL FGWRITE'("DIGOUT6",VL%(),"C.RAW.INT","NT")
5370 ' LP3 EMPTY FLOW-THROUGH CELL
5380 CALL BGWRITE'("LPARRAY","DIGOUT4",1,55,"NT","TASK4")
5390 ' SET PUMP STROKE INTERVAL
5400 CALL INTON'(250,"MIL")
5410 CALL BGGO'("NT","")
5420 STAT%=10
5430 CALL BGSTATUS'("TASK2",STAT%)
5440 IF STAT%<>0 THEN GOTO 5430
5450 ' DEACTIVATE PUMP LP1
5460 VL%(0)=0
5470 CALL FGWRITE'("DIGOUT2",VL%(),"C.RAW.INT","NT")
5480 ' TURN ON AIR FLOW FOR 10 SECONDS DURING RINSE
5490 GOSUB 6340
5500 ' FILL FLOW-THROUGH CELL WITH 18 ML USING PUMP LP2
5510 CALL BGWRITE'("LPARRAY","DIGOUT3",1,33,"NT","TASK3")
5520 CALL INTON'(250,"MIL")
5530 CALL BGGO'("NT","")
5540 STAT%=10
5550 CALL BGSTATUS'("TASK3",STAT%)
5560 IF STAT%<>0 THEN GOTO 5550
5570 ' ACTIVATE BOTH LP2 AND LP3 TO PUMP EQUAL VOLUMES
5580 CALL BGWRITE'("LPARRAY","DIGOUT3",1,80,"NT","TASK5")
5590 CALL BGWRITE'("LPARRAY","DIGOUT4",1,70,"NT","TASK6")
5600 CALL INTON'(250,"MIL")
5610 CALL BGGO'("NT","")
5620 STAT%=10
5630 CALL BGSTATUS'("TASK6",STAT%)
5640 IF STAT%<>0 THEN GOTO 5630
5650 ' TURN OFF STIR FUNCTION
5660 VL%(0)=1
5670 CALL FGWRITE'("DIGOUT6",VL%(),"C.RAW.INT","NT")

```

```

5680 ' DEACTIVATE PUMP LP3
5690 VL%(0)=0
5700 CALL FGWRITE'("DIGOUT4",VL%(),"C.RAW.INT","NT")
5710 STAT%=10
5720 CALL BGSTATUS'("TASK5",STAT%)
5730 IF STAT%<>0 THEN GOTO 5720
5740 ' DEACTIVATE PUMP LP2
5750 VL%(0)=0
5760 CALL FGWRITE'("DIGOUT3",VL%(),"C.RAW.INT","NT")
5770 CALL BGCLEAR
5780 CALL INTOFF
5790 STAT%=10:RETURN
5800 ' SUBROUTINE TO TRANSFER 50 ML FROM RESERVOIR TO IMPACTOR
5810 GOSUB 5140
5820 CALL BGWRITE'("LPARRAY","DIGOUT2",1,66,"NT","TASK11")
5830 CALL INTON'(250,"MIL")
5840 CALL BGGO'("NT","")
5850 STAT%=10
5860 CALL BGSTATUS'("TASK11",STAT%)
5870 IF STAT%<>0 THEN GOTO 5860
5880 ' DEACTIVATE LP1
5890 CALL INTOFF
5900 VL%(0)=0
5910 CALL FGWRITE'("DIGOUT2",VL%(),"C.RAW.INT","NT")
5920 RETURN
5930 ' LP3 EMPTY FLOW-THROUGH CELL
5940 CALL BGWRITE'("LPARRAY","DIGOUT4",1,50,"NT","TASK4")
5950 ' TURN ON STIRRING OF POLAROGRAPHIC CELL DURING RINSE
5960 VL%(0)=0
5970 CALL FGWRITE'("DIGOUT6",VL%(),"C.RAW.INT","NT")
5980 ' SET PUMP STROKE INTERVAL
5990 CALL INTON'(250,"MIL")
6000 CALL BGGO'("NT","")
6010 STAT%=10
6020 CALL BGSTATUS'("TASK4",STAT%)
6030 IF STAT%<>0 THEN GOTO 6020
6040 ' FILL FLOW-THROUGH CELL WITH 18 ML USING PUMP LP2
6050 CALL BGWRITE'("LPARRAY","DIGOUT3",1,33,"NT","TASK3")
6060 CALL INTON'(250,"MIL")
6070 CALL BGGO'("NT","")
6080 STAT%=10
6090 CALL BGSTATUS'("TASK3",STAT%)
6100 IF STAT%<>0 THEN GOTO 6090
6110 ' ACTIVATE BOTH LP2 AND LP3 TO PUMP EQUAL VOLUMES

```

```

6120 CALL BGWRITE'("LPARRAY","DIGOUT3",1,75,"NT","TASK5")
6130 CALL BGWRITE'("LPARRAY","DIGOUT4",1,60,"NT","TASK6")
6140 CALL INTON'(250,"MIL")
6150 CALL BGGO'("NT","")
6160 STAT%=10
6170 CALL BGSTATUS'("TASK6",STAT%)
6180 IF STAT%<>0 THEN GOTO 6170
6190 ' TURN OFF STIR FUNCTION
6200 VL%(0)=1
6210 CALL FGWRITE'("DIGOUT6",VL%(),"C.RAW.INT","NT")
6220 ' DEACTIVATE PUMP LP3
6230 VL%(0)=0
6240 CALL FGWRITE'("DIGOUT4",VL%(),"C.RAW.INT","NT")
6250 STAT%=10
6260 CALL BGSTATUS'("TASK5",STAT%)
6270 IF STAT%<>0 THEN GOTO 6260
6280 ' DEACTIVATE PUMP LP2
6290 VL%(0)=0
6300 CALL FGWRITE'("DIGOUT3",VL%(),"C.RAW.INT","NT")
6310 CALL BGCLEAR
6320 CALL INTOFF
6330 STAT%=10:RETURN
6340 ' TURN ON AIR FLOW FOR 10 SECONDS DURING RINSE
6350 VL!(0)=1.5
6360 CALL FGWRITE'("ANOUT1",VL!(),"C.VOLTS","NT")
6370 VL%(0)=1
6380 CALL FGWRITE'("DIGOUT8",VL%(),"C.RAW.INT","NT")
6390 TIMER ON:RINSETIME=10
6400 ON TIMER (1) GOSUB 6420
6410 IF RINSETIME>0 THEN GOTO 6410 ELSE 6440
6420 RINSETIME=RINSETIME-1
6430 RETURN
6440 VL%(0)=0
6450 CALL FGWRITE'("DIGOUT8",VL%(),"C.RAW.INT","NT")
6460 TIMER OFF
6470 RETURN
6480 CLS:LOCATE 10,1:PRINT "THE RESPONSE TO THE COPPER INTERNAL
      STANDARD WAS MUCH LOWER THAN EXPECTED. THE PREVIOUS TEST
      RESULTS ARE INVALID. IF THE PROBLEM PERSISTS DURING
      SUBSEQUENT TESTS, PLEASE CHECK THE OPERATING MANUAL FOR
      POSSIBLE CAUSES"
6490 ' ROUTINE TO INITIATE 15 MERCURY DROP DISPENSE/DISLODGE
      CYCLES TO ENSURE COLUMN CONTINUITY
6500 FOR J=1 TO 15

```

```

6510 VL%(0)=0
6520 CALL FGWRITE'("DIGOUT7",VL%(),"C.RAW.INT","NT")
6530 VL%(0)=1
6540 CALL FGWRITE'("DIGOUT7",VL%(),"C.RAW.INT","NT")
6550 FOR K=1 TO 2000:NEXT K
6560 NEXT J
6570 LOCATE 20,20:PRINT "PRESS ANY KEY TO CONTINUE ....."
6580 IF INKEY$="" THEN GOTO 6580
6590 CALL KDINIT
6600 CLEAR
6610 GOTO 10
6620 CLS:LOCATE 1,1:FOR J=1 TO 10:PRINT "*****";:NEXT J
6630 TITLE$="LEAD-IN-AIR MONITOR"
6640 LOCATE 2,1:PRINT TAB(40-LEN(TITLE$)/2)TITLE$
6650 LOCATE 3,1:FOR J=1 TO 10:PRINT "*****";:NEXT J
6660 TITLE$="FLUID HANDLING OPTIONS"
6670 LOCATE 9,1:PRINT TAB(40-LEN(TITLE$)/2)TITLE$
6680 TITLE$="-----"
6690 LOCATE 10,1:PRINT TAB(40-LEN(TITLE$)/2)TITLE$
6700 LOCATE 12,1
6710 PRINT TAB(25) "(1) MAIN RINSE"
6720 PRINT TAB(25) "(2) FILL IMPACTOR"
6730 PRINT TAB(25) "(3) EMPTY IMPACTOR"
6740 PRINT TAB(25) "(4) EMPTY POLAROGRAPHIC CELL"
6750 PRINT TAB(25) "(5) EXIT TO MAIN MENU"
6760 PRINT:PRINT:INPUT "ENTER YOUR CHOICE";CHOICE
6770 ON CHOICE GOSUB 5290, 5800, 5930, 6790, 410
6780 GOTO 410
6790 CALL BGWRITE'("LPARRAY","DIGOUT4",1,50,"NT","TASK6")
6800 CALL INTON'(250,"MIL")
6810 CALL BGGO'("NT","")
6820 STAT%=10
6830 CALL BGSTATUS'("TASK6",STAT%)
6840 IF STAT%<>0 THEN GOTO 6830
6850 ' DEACTIVATE PUMP LP3
6860 VL%(0)=0
6870 CALL FGWRITE'("DIGOUT4",VL%(),"C.RAW.INT","NT")
6880 CALL INTOFF
6890 CALL BGCLEAR
6900 GOTO 6620
6910 BEEP:BEEP:BEEP
6920 ON KEY (5) GOSUB 7000
6930 KEY (5) ON
6940 LOCATE 20,18:PRINT "PRESS F5 TO ABORT UNATTENDED OPERATION"

```

```
6950 TIMER ON:WT=30
6960 ON TIMER (1) GOSUB 6980
6970 IF WT>0 THEN GOTO 6970 ELSE 3650
6980 WT=WT-1
6990 RETURN
7000 KEY (5) OFF
7010 LOCATE 20,15:PRINT "UNATTENDED OPERATION HAS BEEN MANUALLY
      ABORTED"
7020 QQ=1
7030 RETURN
```

APPENDIX E
OPERATING MANUAL FOR THE LEAD-IN-AIR MONITOR

E.1 SETTING UP THE LEAD-IN-AIR MONITOR

Unpacking Instructions

Parts List

The lead-in-air monitor consists of the following major components:

<u>Qty</u>	<u>Description</u>
1	Module 1. Contains liquid pumps, electrolyte reservoir, impactor, nitrogen handling components, digital logic board, and compression fittings.
1	Module 2. Contains air pump, cartridge filter, mass flow controller, recirculation valve, and compression fittings.
1	EG&G Model 303A static mercury drop electrode system with Model 305 stirrer.
1	EG&G Model 264A polarograph control unit.
1	PC Brand Model 286/25 computer with monochrome monitor and keyboard.
1	10 liter capacity polypropylene wide mouth waste container (Fisher 02-961-65A) with attached teflon bulkhead 1/4 inch pipe connectors.
1	Stainless steel utility cart (McMaster-Carr 2544T4).
1	22 inch long, 1/4 inch O.D. teflon tube for connecting Module 1 waste outlet to waste container.
5	6 ft. 120 VAC power cords.

The Model 303A, Model 264A, Keithley 575, and computer each have their own instruction manuals and set of accessories. Refer to these manuals for specific operating requirements and maintenance.

The following accessories are also included with the system:

<u>Qty</u>	<u>Description</u>
2	10 liter capacity polypropylene wide mouth containers (Fisher 02-961-65A) for preparation and storage of electrolyte.
1	3 lbs. triple-distilled mercury.
1	34 liter cylinder of ultra-high purity nitrogen.
1	1/4 inch tubing grooving tool.
1	1 liter capacity polypropylene volumetric flask (Fisher 10-198-50F) for preparing and storage of calibration check standard.
1	15 ml and 25 ml pipettes and squeeze bulb.
1	Spare cartridge filter.
1	39 inch teflon-lined sampling probe and fittings.
1	EG&G Model 9103 Ag/AgCl filling solution for reference electrode.
1	Replacement teflon fittings and tubing.
1	Thermocouple readout (Omega HH-25KF Type K), flexible connector, and probe (CASS-14G-24).
1	Parallel printer cable.
1	1 ml positive displacement precision pipette with disposable tips.
3	Spare 25A solid state relays.

Polarograph Setup

Most of the lead-in-air monitor's components can be handled and transported without special precautions. The Model 303A polarograph, however, is a sensitive piece of equipment and requires special attention with regard to its handling and use.

During its use, the polarograph is housed within Module 1 and is connected to the various Module components. As long as the system remains fairly level, the unit may be safely moved from one location to another. If the equipment must be shipped, however, the polarograph should be removed from the module and prepared for shipping. Instructions for preparing the polarograph for shipping are detailed in Section E-5.

Following its shipping, the polarograph can be setup for operation by the following procedures:

- 1) Unpack the Model 303A from its shipping container and place it on a stable, level surface. If available, use of a ventilated fume hood is recommended.
- 2) Unpack the Model 264A control unit and place it next to the Model 303A. Using the 25 pin D-sub ribbon cable (coded AA-JJ), connect the Model 264A to the Model 303A. Connecting ports are located at the rear of each unit. Connect the Model 264A to a 120 VAC power source.
- 3) Unpack and install the glass capillary in the Model 303A electrode support block according to the procedures outlined in the Model 303A manual.
- 4) Carefully following the procedure described in the Model 303A manual, fill the Model 303A reservoir with approximately 2 kg mercury. To ensure accurate test results, use only high-purity, triply-distilled or better analytical grade mercury. The mercury supplied by Bethlehem Apparatus Corp. has been found suitable for this purpose and is recommended.

Should you encounter any difficulty in filling the mercury reservoir or attaining continuity in the glass capillary, contact the manufacturer for assistance.

- 5) . Unpack the polarograph reference electrode. Using the technique outlined in the Model 303A manual, fill the glass sleeve of the reference electrode to within 1 cm of capacity using the supplied Ag/AgCl solution. Make sure the sleeve is free of air bubbles before its installation. If necessary, a very small quantity of petroleum jelly can be applied to the sleeve's external o-ring to ease its insertion into the electrode support block.
- 6) Add approximately 10 to 15 ml of D.I. water to a teflon static cell and install it within the Model 303A. This immerses the electrodes in the water and prevents the reference electrode from drying out. The static cell will later be replaced by the flow-through cell.
- 7) Unpack Module 1 and place it on the utility cart's top shelf in the location shown in Figure E-1. Make sure its door can open and close freely without hitting the upraised edge of the cart.
- 8) Disconnect the ribbon cable from the Model 303A. Carefully place the Model 303A inside Module 1 in the location shown in Figure E-2. Connect the 25 pin D-sub cable (attached to the digital logic board) to the rear of the Model 303A. Also attach the 1/4 inch flexible tube (which supplies nitrogen purge gas) to the hose-barb fitting at the rear of the Model 303A.

Setup of Remaining Components

- 1) Place the Model 264A on the cart's second shelf in the position indicated in Figure E-1. Connect the Model 264A ribbon cable to the rear of Module 1 as coded.
- 2) Unpack Module 2 and place it on the cart's lower shelf in the position shown in Figure E-1.
- 3) Unpack the computer and place it next to the Model 264A on the second shelf. Place the monochrome monitor on the cart's top shelf next to Module 1 and connect the monitor cable to the computer's labeled port. Attach the keyboard cable to the computer port (marked K.B.).
- 4) Unpack the Keithley 575 data acquisition and control system and place it on top of the Model 264A.



Figure E-1. Photograph of lead-in-air monitor showing arrangement of components on utility cart.

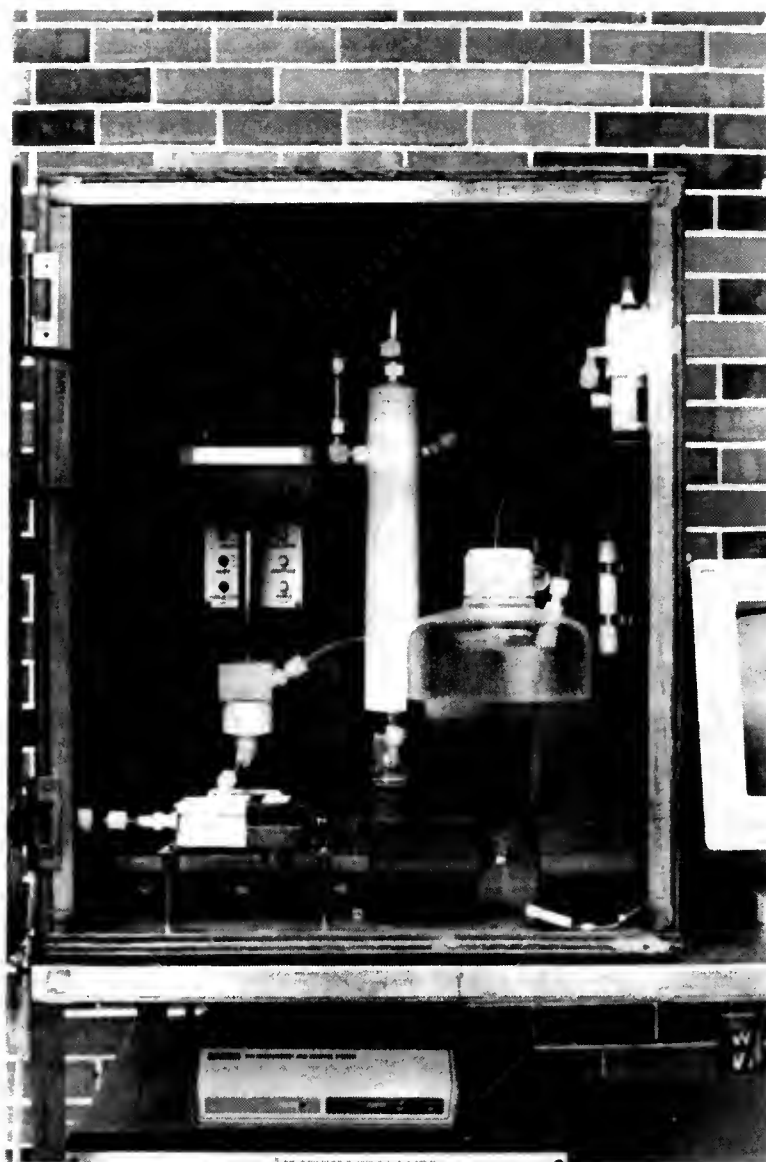


Figure E-2. Photograph of Module 1 interior showing location of components.

- 5) Connect all remaining ribbon cables into the appropriate component ports using the connectors codes as guides. Descriptions of external cable connections are provided in Table E-1.
- 6) Attach power cords to the computer, Module 1, Module 2, and Model 264A. Plug these cords (plus the monitor's power cord) into the power strip installed on the cart's lower shelf.
- 7) Unpack the 10 liter polypropylene waste container and place it on the the cart's bottom shelf next to the power strip. Connect the waste container to the Module 1 waste outlet using the supplied 22 inch long 1/4 inch O.D. teflon tube.

Note: To prevent fluid leakages, all teflon tubes in the lead-in-air monitor have been scored using the supplied tubing grooving tool. Should tubing ever need to be replaced, refer to the grooving tool's manual for instructions concerning its use.

Use the following procedures to ensure that tubing connectors are properly installed.

- 1) Slide the ferrule nut past the groove on the teflon tube.
 - 2) Insert the end of the tube into the fitting until it bottoms out.
 - 3) Slide the ferrule nut to the fitting and hand tighten.
 - 4) Pull on the tube slowly until it catches on the grooved edge.
 - 5) Hand tighten the ferrule an additional 1/2 to 3/4 of a turn. Should a leak in the fitting occur during its use, repeat the above steps. If the leak persists, replace the fitting and the teflon tubing.
- 8) Connect the Module 1 air outlet to the Module 2 air inlet using the supplied 1/2 inch O.D. polypropylene tube.
 - 9) Install a nitrogen cylinder (with attached regulator) within its support block inside Module 1. Connect the 1/4 inch flexible tube to the regulator outlet. Open the regulator valve by turning it fully counterclockwise.
 - 10) Unpack the 5 liter teflon reservoir and place it within its Module 1 support block. As shown in Figure E-2, make sure the fluid level

Table E-1
Description of External Cable Connections

<u>Cable Code</u>	<u>Connection</u>
AA (Module 1)	JJ (Model 264A)
BB (Module 1)	DD (Model 575)
CC (Model 575)	II (Model 264A)
DD (Model 575)	BB (Module 1)
EE (Model 575)	KK (Module 2)
FF (Model 575)	LL (Computer)
GG (Model 575)	HH (Model 264A)
HH (Model 264A)	GG (Model 575)
II (Model 264A)	CC (Model 575)
JJ (Model 264A)	AA (Module 1)
KK (Module 2)	EE (Model 575)
LL (Computer)	FF (Model 575)

switch is positioned within the cutout of the support block. Plug in the level switch quick-disconnect wires. Attach the 1/4 inch O.D. flexible tygon tube to the hose barb connector of the glass-wool packed drying tube. Lastly, attach the 1/4 inch teflon tube to the inlet fitting of liquid pump LP1.

- 11) To fill the electrolyte reservoir, first remove the container's outer and inner caps and place them on a clean surface. Using the supplied polypropylene funnel to prevent spillage, fill the reservoir with the 0.5M HNO₃/200 ppb copper electrolyte. Procedures for preparing the electrolyte are outlined in section E . Remove the funnel and replace the two teflon caps.
- 12) Remove the static cell from the polarograph. To do this, support the cell with one hand and swing the spring-loaded support plate out of position. Then lower the teflon cup straight down until the electrodes are free from the cup. Discard the cup's contents and store the cup for later use.
- 13) Unpack the teflon flow-through cell and place the supplied magnetic stirring bar inside it. Rinse the cell twice with D.I. water. Install the cup under the electrode support block and hold in place. While holding the cell in position, slide the Model 305 stirrer under the cell until the cell is centered over the stirrer. The stirrer supports the cell in place. Connect the cell's outlet tube to the inlet of liquid pump LP3. Note that the polarographic will have to shifted back and forth slightly to properly attach the tube fittings.
- 14) Using the supplied 15 ml pipette and squeeze bulb, introduce 15 ml of D.I. water into the flow-through cell through the open connector of the electrode support block. If the liquid is observed to flow down to liquid pump LP2, it indicates a leak in the tubing connectors.
- 15) Attach the teflon tube from liquid pump LP2 to the inlet tube on the electrode support block. See Figure E-3 for a view of the flow-through cell and the support block connector.
- 16) Set the Model 303A front panel controls as indicated in Table E-2. Once these switches are set, they need not be changed during use of the lead-in-air monitor.
- 17) Set the Model 264A front panel controls as outlined in Table E-2. Make sure that values are set properly. Once the values are set, they need not be changed during normal use of the instrument.

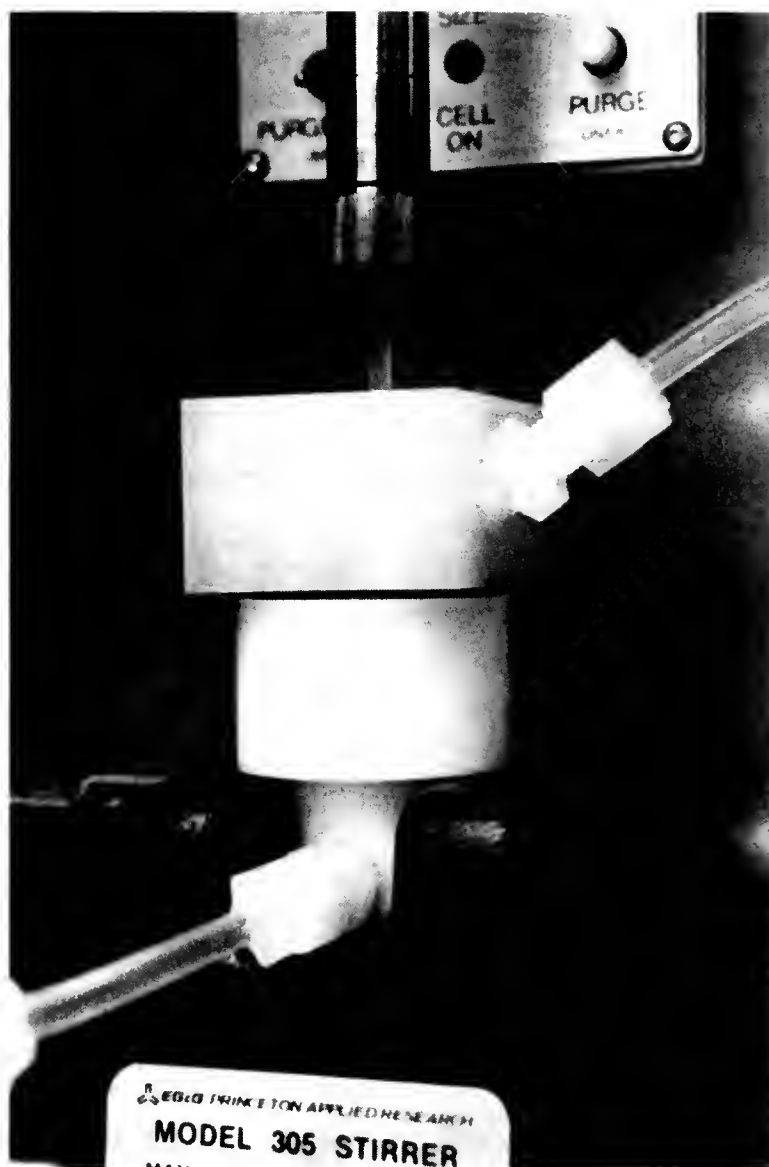


Figure E-3. Photograph of flow-through polarographic cell showing location of inlet and outlet fittings.

Table E-2
Standard Settings for Polarograph and Analyzer

Model 303A Polarograph

Drop Enable = On

Mode = HMDE

Drop Size = M

Purge Time (min) = 2

Model 264A Analyzer

Initial Potential (Volts) = - 0.60

Final Potential (Volts) = +0.20

Scan Rate (mV/sec) = 10

Meter = Current

Current Range = 20 μ A

Filter (DC Only) = Off

Output Offset = Straight Up

Purge Time (minutes) = 0

Deposition Time (seconds) = 180

Equil Time = 15 sec

Drop Time = 0.5 sec

Pulse Height = 50 mV

Replication = 1

Analysis Mode = Diff Pulse Stripping

- 18) With the power strip off, plug in all power cords. Then with the power still off, turn on the Model 264A (rear panel), Kiethley 575 (front panel), monitor (front panel), and computer (front panel). These components will thus power up when the power strip is turned on.

This completes the initial setup of the lead-in-air monitor. Proceed to the following section to perform initial checks of the system.

E.2 PREPARATION OF STANDARD SOLUTIONS

Electrolyte Preparation

The electrolyte used in the lead-in-air monitor consists of 0.5M HNO₃ with a 200 ppb internal copper standard. The accuracy and detection limit of the monitor depends on the quality of the prepared electrolyte. Preparation of the electrolyte should be performed in a clean work area and the following steps performed as carefully as possible.

- 1) To a clean, acid-washed 10 liter polypropylene container, add approximately 5 liters of D.I. water. The exact volume added is not critical.

Note: Use the best quality water available. Distilled water manufactured by Zephyrhills Spring Water Co. has been found satisfactory for this purpose and is available from most supermarkets. Other brands may also be used as long the water passes the "Solution Background Check" outlined in Section E.3.

- 2) To the container, add 300 ml of concentrated nitric acid. The use of trace metal grade (Fisher A509-212) is recommended for this purpose. Do not use reagent grade nitric acid as its impurity content may be unacceptably high.
- 3) Add 2 ml of 1000 ppm copper standard (Fisher SC194-500) to the container. The use of the supplied positive displacement pipette with disposable tips is recommended to ensure accurate results. Refer to the pipette's accompanying manual to ensure its proper use.
- 4) Add distilled water to the container until the 10 liter fluid level is reached. Cap the container tightly and mix the contents thoroughly. Label and date the container. This solution may be stored up to one month in this container. After one month, the contents should be disposed of properly and a new solution prepared.
- 5) To fill the Module 1 reservoir, remove the reservoir's outer and inner caps and place them on a clean surface. Using the supplied polypropylene funnel to minimize spillage, fill the

reservoir with the electrolyte. Immediately wipe up any spills which may occur. Remove the funnel and replace the two teflon caps.

100 ppb Lead Standard Preparation

The 100 ppb lead standard is used to verify the accuracy of the lead-in-air monitor's analysis section. Prepare the lead standard in a clean work area free from possible lead contamination. To ensure accurate test results, perform the following steps carefully:

- 1) To an acid-washed, 1000 ml volumetric polypropylene flask (Fisher 10-198-50F), add approximately 500 ml of distilled water. The exact volume of water added is not critical. Use the same quality water used for preparation of the electrolyte.
- 2) To the container, add 30 ml of trace metal grade nitric acid.
- 3) Using a clean 25 ml pipette and squeeze bulb, add to the container 25 ml of certified 1000 ppb lead standard (Fisher SL21-500).
- 4) Add distilled water to the container until the 1000 ml level is reached. Cap the container tightly and mix the contents thoroughly.

The resulting solution is a 25 ppm lead standard solution which will later be diluted to prepare the 100 ppb lead standard. Label and date the container. If this 25 ppm container is refrigerated, it may be stored for several months before a fresh solution need be prepared.

- 5) To an separate acid-washed, 1000 ml volumetric polypropylene flask, add approximately 500 ml of the 0.5M HNO₃/200 ppb copper electrolyte prepared earlier. The exact volume of electrolyte added is not critical.
- 6) Using the 1 ml positive displacement pipette with a fresh tip, add 4 ml of the 25 ppm lead standard solution to the container.

- 7) Add 0.5M HNO₃/200 ppb copper electrolyte to the container until the 1000 ml fluid level is achieved. Cap the container and mix the contents thoroughly.

The resulting concentration of this solution is 100 ppb lead standard in 0.5M HNO₃ with a 200 ppb copper standard. Label and date the container. The solution can then be used to perform the calibration check which will be outlined in Section 3.3. The shelf life of this solution is approximately two weeks. After that time period, its contents should be disposed of properly and a fresh solution prepared.

E.3 INITIAL SYSTEM CHECKS

The following system checks should be performed whenever the system has been setup following its shipping. These system checks can also be performed at any time to verify the working condition of the lead-in-air monitor.

Instrument Status

Turn on the power strip. Following the autoload of the main computer program, verify the following instrument conditions:

Kiethley 575: Red power light is **on**

Model 303A: Green "drop dispenser" light is **on**
Red "cell on" light is **on**

Model 264A: Digital panel meter reads **0.000 ± 0.010 volts**
The meter typically displays a +0.004 V value

Red "standby" light is **on**. All other lights on the Model 264A should be **off**.

Computer: Yellow "turbo" light is **on**
Green "power" light is **on**

Monitor: Lead-in-air monitor's **Main Menu** appears

If the system passes these initial checks, procede to the next section. Otherwise consult the troubleshooting guide in Section E-6 for possible explanations.

Solution Background Check

The following procedure is useful for verifying the overall operation of instrument.

- 1) Turn on the power strip to power the system components.
On the monitor, the following menu will appear:

Main Menu

- 1) Perform Single Sampling and Analysis
 - 2) Unattended Sampling and Analysis
 - 3) Analyze Solution Only
 - 4) View or Print Previous Test Results
 - 5) Fluid Handling Options
 - 6) Exit Program
- 2) From this main menu, select option 5 - Fluid Handling Option.
- 3) A menu of fluid handling options will appear:

Fluid Handling Options

- 1) Main Rinse
- 2) Fill Impactor
- 3) Empty Impactor
- 4) Empty Polarographic Cell
- 5) Exit to Main Menu

From this menu, select option 1 - Main Rinse.

Caution: Make sure the inlet plug is removed from the Module 1 air inlet section.

- 4) With the Module 1 door open, observe the sequence of the main rinse routine. Liquid pumps LP1 and LP3 should simultaneously fill the impactor and empty the polarographic cell, respectively. Check the liquid fittings for possible leaks. Once the impactor is filled, the air pump should activate for 10 seconds to thoroughly rinse the containers walls. Liquid pump LP2 will then activate to fill the flow-through cell. Once filled, LP3 and LP2 will operate concurrently until the impactor is empty. The flow-through cell should now contain approximately 15-18 ml of electrolyte. The exact volume present is not critical.

You will then be returned to the Main Menu.

- 5) If the system is being checked following its shipment or transport, repeat steps 2 through 4. Otherwise proceed to step 6.
- 6) From the main menu, select option 3 - Analyze Solution. At the prompt, enter an appropriate description for this test.

Analysis will first begin with a 3 minute nitrogen purge. Adjust the rotameter until the flowrate reads 0.03 to 0.05 lpm. Following the purge cycle, the 3 minute deposition phase will begin as indicated by the monitor and the **deposit** light on the Model 264A front panel. A 15 second equilibrium time will then take place indicated by the **equilibrate** light. An 80 second analysis time will then occur indicated by the **scan** light and the computer monitor. During the analysis phase, the 264A panel meter displays the measured scan currents.

- 7) Following a brief calculation phase, the test results will be displayed on the monitor. Record the value of the liquid lead concentration (ppb) and the magnitude of the lead and copper peak heights (volts). Repeat steps 6 and 7 at least two more times.

The average of the reported liquid lead concentrations during three analysis should be less than 20 ppb. As explained in Chapter 5 of the text, this response is normally due to the presence of hydronium ions rather than background lead content of the solution itself. If the average calculated lead concentration is higher than 20 ppb, consult the troubleshooting guide for assistance.

The average voltage corresponding to the 200 ppb copper content of the electrolyte should be approximately **-1.600 V \pm 50%**.

This completes the instrument background check. Proceed to the following section to verify the instrument's lead detection capabilities.

Lead Calibration Check

To verify the instrument's ability to accurately quantify a known liquid lead concentration, perform the following steps:

- 1) Turn on the power strip to power the system components.
At the Main Menu, select option **5** - Fluid Handling Options.
- 2) Select option **1** to perform a Main Rinse.
- 3) At the completion of the rinse routine, you will be returned to the Main Menu. Select option **5** - Fluid Handling Options.
- 4) Select option **4** - Empty Polarographic Cell.
- 5) Loosen the ferrule nut at the electrode support inlet fitting (Figure E-3) and remove the teflon tube.
- 6) Using the supplied 15 ml pipette and squeeze bulb, dispense 15 ml of the prepared 100 ppb lead standard solution through the inlet fitting into the flow-through cell. Reattach the inlet tube.
- 7) Select option **5** - Exit to Main Menu.
- 8) From the Main Menu, select option **3** - Analyze Solution Only. At the prompt, enter an appropriate description for this test.
- 9) Following the analysis, the test results will be displayed on the monitor. Record the values of the liquid lead concentration (ppb) and the magnitude of the lead and copper peak heights (volts). Steps **8** and **9** should be repeated at least two additional times.

The average of the reported liquid lead concentrations during three analyses should be equal to **100 ppb \pm 5%**. If the reported concentration is outside this range, consult the troubleshooting section for possible causes.

The average voltage corresponding to the 200 ppb copper content of the electrolyte should be approximately **-1.600 V \pm 50%**.

- 10) From the Main Menu, select option **5** - Fluid Handling Option. From the fluid handling menu, select option **1** - Main Rinse.

E.4 OPERATING INSTRUCTIONS

Presampling Checklist

Once the lead-in-air monitor has been setup and the initial system checks completed, air sampling can be performed. Prior to the sampling, it is recommended that the following steps be performed.

- 1) Inspect the electrolyte reservoir and verify that the available supply is sufficient for the intended tests. If necessary, fill the reservoir before use. Also make sure that the nitrogen tank pressure is at least 50 psig. If not, replace the tank using the procedures outlined in section E.5.
- 2) Make sure that the 10 liter waste container is not more than half full. As a conservative guideline, the waste container should be emptied each time the electrolyte reservoir is filled.

Note: The waste fluid consists of nitric acid with significant quantities of lead and copper. Handle and dispose of this solution properly.

The waste container will also contain small amounts of mercury. Rather than pouring this mercury out each time the container is emptied, it is recommended that the mercury be allowed to accumulate at the bottom of the waste container. When a significant quantity of mercury accumulates (over several hundred test runs), it may be collected and shipped to Bethlehem Apparatus Corp. to be refined and reused.

- 3) If static sampling is to be performed, make sure the following components are available:
 - a. Temperature gauge, flexible connector, and temperature probe.
 - b. 6 inch long, 1 inch O.D. stainless steel inlet nozzle.
 - c. Extension cord of sufficient length, rated for 20A service.
 - d. Tools for attaching inlet nozzle to Module 1 bulkhead inlet fitting.
 - e. Notebook

- 4) If duct sampling is to be performed, the following components are required:
 - a. Temperature gauge, flexible connector, and temperature probe.
 - b. Stainless steel inlet nozzles of 3/16, 1/4, and 1/2 inch diameter with accompanying 1 inch female connector.
 - c. Flexible teflon-lined probe.
 - d. Stainless steel standard pitot tube (Dwyer 160-24) with two six foot section of 3/16 inch I.D. tygon tubes.
 - e. 0-3 inch H₂O manometer gauge (Dwyer Mark II Model 25) or equivalent pressure gauge.
 - f. Extension cord of sufficient length, rated for 20A service.
 - g. Tools for attaching nozzles and probe.
 - h. Notebook

Sampling and Analysis Procedures

Still Air Sampling

- 1) Attach the stainless steel nozzle to the bulkhead fitting at the top of Module 1 and tighten the compression fitting. It is recommended that this inlet be covered with a plastic bag until just prior to air sampling.
- 2) Move the monitor to the desired point of sampling.
- 3) Using the extension cord, connect the power strip to an available 120 VAC, 20A power supply.
- 4) With the temperature gauge, measure and record the air temperature (°F) at the intended point of sampling.
- 5) Turn on the power strip to power the system components.
- 6) At power up, the computer program loads and executes automatically to present the main menu:

Main Menu

- 1) Perform Single Sampling and Analysis
- 2) Unattended Sampling and Analysis
- 3) Analyze Solution Only
- 4) View or Print Previous Test Results
- 5) Fluid Handling Options
- 6) Exit Program

Select either option **1** or **2** as desired. Single point sampling is chosen if the user wants to perform single point analysis at various sampling locations within the plant. After each test, the user is returned to the Main Menu for the next selection. The unattended sampling and analysis option allows the user to perform repeated sampling and analysis tests at a single sampling without input from the operator.

- 7) At the prompt, enter the gas temperature measured earlier.
- 8) The user is then prompted for the measured gas velocity pressure. For the still (static) air sampling, enter a **0** value.
- 9) At the prompt, enter an appropriate description for this test. This description may contain up to 30 characters and will be saved on the computer's hard drive along with the test results.
- 10) Press the spacebar to begin air sampling. Sampling will be performed for 15 minutes at a flowrate of 15 alpm. During the test, the sampling time remaining will be displayed on the monitor.
- 11) When the air sampling is completed, the collected lead is quantified and the test results displayed on the monitor. These results are automatically saved to the computer's hard drive for later inspection.

If operating in Single Sampling and Analysis mode, press any key to return to the Main Menu.

If operating in Unattended Sampling and Analysis mode, the test results will be displayed for 30 seconds before beginning another sampling and analysis cycle. During this display period, the user is prompted to press function key **F5** if he desires to abort the

unattended operation. Pressing F5 returns the user to the main menu for the next selection.

- 12) Upon completing the desired tests, the lead-in-air monitor can be shutdown. To do this, simply exit the program and turn off the system's power strip.

Note: Should a power interruption occur during the sampling or analysis cycle, the computer will automatically reboot the program and the current test information will be lost. If the interruption occurred during air sampling, 50 ml of the electrolyte will remain in the impactor. This volume must be removed prior to the next test or the flow-through cell will overflow. To empty the impactor following a power outage, perform the following steps:

- 1) At the Main Menu, select option 5 - Fluid Handling Options.
- 2) At the Fluid Handling Menu, select option 3 - Empty Impactor. Liquid pumps LP2 and LP3 will be sequenced to transfer the impactor's contents to the waste container.

The user can then return to the Main Menu and repeat the test series.

Ducted Air Sampling

As discussed in Chapter 4, accurate collection and transport of particulates from ducted airstreams requires that special sampling considerations be observed. Alternatives to the supplied sampling nozzles and probe have been presented and can be investigated. This section will outline procedures necessary for use of the supplied duct sampling equipment.

- 1) Position the lead-in-air monitor as close to the intended point of sampling as possible. Access to ducted airstreams may require that special support platforms be designed and constructed to accommodate the monitor.
- 2) Using the extension cord, connect the power strip to an available 120 VAC, 20A power supply.

- 3) Select the point of sampling based on the duct configuration and access availability. Consider port locations at 90° duct bends where the sampling probe can be directed into the oncoming airstream. For single point sampling, it is better to sample at the centroid rather than the midpoint of the duct's cross-section.
- 4) With the temperature gauge, measure and record the air temperature (°F) at the intended point of sampling.
- 5) Prepare the standard pitot tube for velocity measurements. Connect the total pressure port of the pitot tube to the high side of the manometer using one section of the 3/16 inch I.D. flexible tubing. Using the other piece of tubing, connect the static port of the pitot tube to the low side of the manometer. Zero and level the manometer at this time.
- 6) At the desired point of sampling, direct the pitot tube into the oncoming flowstream and record the resulting velocity pressure (inches of water) indicated on the manometer.
- 7) Turn on the power strip to provide power to the lead-in-air monitor's components.
- 8) At power up, the computer program loads and executes automatically to present the main menu:

Main Menu

- 1) Perform Single Sampling and Analysis
- 2) Unattended Sampling and Analysis
- 3) Analyze Solution Only
- 4) View or Print Previous Test Results
- 5) Fluid Handling Options
- 6) Exit Program

Select either option **1** or **2** as desired. Single point sampling is chosen if the user wants to perform single point analysis at various sampling locations within the plant. After each test, the user is returned to the Main Menu for the next selection. The unattended sampling and analysis option allows the user to perform repeated sampling and analysis tests at a single sampling without input from the operator.

- 9) At the prompt, enter the gas temperature measured earlier.
- 10) As requested, enter the measured velocity pressure recorded earlier.
- 11) A calculation is then made of the gas' velocity and the user is notified of the recommended nozzle size required to achieve isokinetic sampling under these conditions. Select an available nozzle diameter nearest to this size and enter the diameter into the program. The program will later adjust the sampling flowrate to provide isokinetic sampling through the selected nozzle.
- 12) At the prompt, enter an appropriate description for this test. This description may contain up to 30 characters and will be saved on the computer's hard drive along with the test results.
- 13) As indicated in Figure 4-1, attach the selected nozzle to the sampling probe then connect the probe to the Module 1 bulkhead inlet. Make sure that all fittings are tight.
- 14) Insert the nozzle and probe through the sampling port. Position the probe so it is directed into the oncoming airstream. Support the probe as needed.
- 15) Press the spacebar to begin the air sampling cycle. Air sampling will be performed for 15 minutes.
- 16) When the air sampling is completed, carefully remove the probe from the duct and support it as needed. When the collected lead is quantified, the test results will be displayed on the monitor. These results are automatically saved to the computer's hard drive for later inspection.

If operating in Single Sampling and Analysis mode, press any key to return to the Main Menu.

If operating in Unattended Sampling and Analysis mode, the test results will be displayed for 30 seconds before beginning another sampling and analysis cycle. During this display period, the user is prompted to press function key **F5** if he desires to abort the unattended operation. Pressing F5 returns the user to the main menu for the next selection.

- 17) Upon completing the desired tests, the lead-in-air monitor can be shutdown. To do this, simply exit the program and turn off the system's power strip.

Note: Should a power interruption occur during the sampling or analysis cycle, the computer will automatically reboot the program and the current test information will be lost. If the interruption occurred during air sampling, 50 ml of the electrolyte will remain in the impactor. This volume must be removed prior to the next test or the flow-through cell will overflow. To empty the impactor following a power outage, perform the following steps:

- 1) At the Main Menu, select option 5 - Fluid Handling Options.
- 2) At the Fluid Handling Menu, select option 3 - Empty Impactor. Liquid pumps LP2 and LP3 will be sequenced to transfer the impactor's contents to the waste container.

The user can then return to the Main Menu and repeat the test series.

Viewing or Printing Previous Test Results

Following each sampling and analysis test, the test results are displayed on the computer monitor for the user's inspection. These results are also automatically saved on the computer's hard drive for later inspection. All test results are stored in the directory C:\K500\RESULTS created for this purpose.

To view previously saved test results, perform the following steps:

- 1) From the Main Menu, select option 4 - View or Print Previous Test Results. The following menu will appear:

Menu of Test Results

- 1) List Existing File Names
- 2) View Archived Test Results
- 3) Print Archived Test Results
- 4) Exit to Main Menu

- 2) If desired, select option 1 to display the directory of existing file names.

Test results are stored under filenames equivalent to the date the test was performed. Multiple test results performed on the same day are saved in the same file. After viewing the file directory, press any key to return to the menu.

- 3) To view a specific saved file, select option **2** - View Archived Test Results. At the prompt, enter the filename you wish to view. Either the mm/dd/yy or mm-dd-yy format is acceptable.

Information displayed for each test includes the test's starting time, test duration, average sampling flowrate, liquid and air lead concentration, and a test description. The monitor will display ten test results at a time. Press the spacebar to view the next screen of test results. When the last test results have been viewed, press any key to return to the view menu.

- 4) Printing previous test results first requires that the user connect the computer to a parallel letter-quality or dot-matrix printer using the supplied parallel printer cable. Once the printer is connected and turned on, select option **3** - Print Archived Test Results.

At the prompt, enter the filename you wish to view. Either the mm/dd/yy or mm-dd-yy format is acceptable.

Information printed for each test includes the test's starting time, test duration, average sampling flowrate, liquid and air lead concentration, and a test description. When the test results have been printed, press any key to return to the view menu.

E.4 MAINTENANCE OF THE LEAD-IN-AIR MONITOR

If normal care is observed during its handling and use, the lead-in-air monitor is expected to require little periodic maintenance. Unless a malfunction occurs, the computer, data acquisition and control system, and the Model 264A control unit should provide extended service without the need for periodic maintenance. At regular intervals, however, it is recommended that the following maintenance be performed to ensure that the monitor functions accurately.

Maintenance of the Reference Electrode

The Model 303A polarograph's reference electrode contains a Ag/AgCl reference solution which provides a stable potential upon which the voltametric measurements are based. Because the reference electrode is immersed in the nitric acid electrolyte, the reference solution will eventually become diluted with time. This may result in a shift in the peak potentials measured during the lead analysis. To ensure accurate test results, it is recommended that the reference solution be replaced at monthly intervals. Procedures for replacing the solution are as follows:

- 1) Turn on the power strip to provide power to the system components.
- 2) At the Main Menu, select option 5 - Fluid Handling Options.
- 3) At the Fluid Handling Menu, select option 4 - Empty Polarographic Cell. When the cell is empty, turn off the power strip.
- 4) Remove the flow-through cell from Module 1. First, loosen the teflon ferrule at the liquid pump LP3 inlet. Rotate the inlet connector slightly to remove the teflon tube from the fitting. While supporting the flow-through cell with one hand, slide the Model 305 stirrer forward until it is free from the cell. Set the stirrer aside. Lower the cell straight down until the electrodes are free from the cup.

Caution: The cell may contain a small quantity of mercury from previous test runs. Collect this mercury for later recycle. The cup can also be cleaned at this time by rinsing it with 0.1M nitric acid, wiping it with a KimWipe, and rinsing it with distilled water.

- 5) Using firm downward pressure, remove the glass electrode from the plastic support block and discard its contents. Inspect the Vycor frit at the end of the glass sleeve. If it appears cracked or damaged, replace it according to the procedures outlined in the Model 303A manual.

Rinse out the electrode with the supplied Ag/AgCl filling solution then fill it to within 1 cm of capacity. Make sure the sleeve is free of air bubbles before proceeding.

- 6) Reinstall the reference electrode within the support block. In necessary, a very small quantity of petroleum jelly may be applied to the sleeve's external o-ring to ease its insertion into the support block.
- 7) Reinstall the flow-through cell, replace the stirrer, and reconnect the teflon tube to the liquid pump LP3 inlet.
- 8) Turn on the power strip to provide power to the system components. At the Main Menu, select option 5 - Fluid Handling Options.
- 9) At the Fluid Handling Menu, select option 1 - Main Rinse. Perform the main rinse routine two times. Return to the Main Menu and exit the program.

This completes the required maintenance of the reference electrode.

Impactor Maintenance

Repeated use of the impactor to collect particulates may result in the accumulation of deposits within the impactor's inner surfaces. It is recommended that these deposits be removed at regular intervals. Experience will dictate the frequency at which this maintenance occurs. For static or recirculation air sampling, it is expected that many tests can be performed before cleaning is necessary. A greater cleaning frequency can be expected if higher aerosol concentrations are routinely sampled.

Cleaning of the impactor requires its removal from Module 1. Refer to Figure 4-1 and perform the following steps:

- 1) Loosen the ferrules of the 3/8 inch union elbow (component 9) and remove the elbow. Loosen and remove the 3 7/8 inch tube (component 8) attached to the elbow.

- 2) Loosen the retaining nut of the 1 inch bulkhead connector (component 4) until the nut has traveled approximately 1 inch on the threads.
- 3) Loosen the teflon ferrule of the impactor liquid inlet and remove the teflon tube.
- 4) Rotate the impactor counter-clockwise to unscrew it from liquid pump LP2. When detached, lift the impactor straight up and tilt it until its bottom fitting is clear of the pump. Lower the impactor until the stainless steel inlet tube is clear of the bulkhead fitting. Remove the impactor from Module 1.
- 5) Begin disassembly of the impactor by unscrewing and removing its teflon housing inlet from the main body. Rinse the interior surfaces of the housing with 0.1M nitric acid and wipe its surfaces with a KimWipe. Rinse the housing with distilled water and set aside to dry. When dry, remove any teflon tape from the bottom pipe fitting and rewrap with two turns of 1/2 wide teflon tape.

Unscrew the impactor stage from the central tube and rinse these components with 0.1M nitric acid. Wipe all surfaces with KimWipes, rinse with distilled water, and set aside to dry.

When all the impactor components are dry, reassemble the impactor and install it within Module 1 by reversing the disassembly procedures. Make sure all fittings are tight to ensure proper seals.

Nitrogen Cylinder Replacement

The supplied nitrogen cylinders have a rated capacity of 34 liters of nitrogen at standard conditions. This supply is sufficient for approximately 200 separate analyses before the cylinder need be replaced. Periodically check the pressure gauge to ensure that the supply pressure has not dropped below 50 psig. To replace the cylinder, perform the following steps:

- 1) Close the regulator valve by turning the valve fully clockwise.
- 2) Remove the flexible tubing from the regulator's hose barb outlet connector. Remove the cylinder from its Module 1 support block.

- 3) Unscrew the regulator and remove it from the empty cylinder. Dispose of the cylinder properly. These cylinders are not rechargeable.
- 4) Screw the regulator into a fresh cylinder and install the cylinder inside Module 1. Attach the flexible tubing and fully open the regulator valve.

E.5 SHIPPING THE LEAD-IN-AIR MONITOR

With the exception of the Model 303A polarograph, most of the lead-in-air monitor's components can be packed and shipped successfully simply by taking normal packing precautions. The procedures necessary to prepare the polarograph for shipping will be outlined in this section. Preparation of other system components will also be discussed.

- 1) Turn on the power strip to power the system components. At the Main Menu, select option 5 - Fluid Handling Option. At the Fluid Handling Menu, select option 4 - Empty Polarograph Cell. When the cell is empty, turn off the power strip.
- 2) Remove the flow-through cell from Module 1. First, loosen the teflon ferrule at the liquid pump LP3 inlet. Rotate the inlet connector slightly to remove the teflon tube from the fitting. While supporting the flow-through cell with one hand, slide the Model 305 stirrer forward until it is free from the cell. Set the stirrer aside. Lower the cell straight down until the electrodes are free from the cup.

Caution: The cell may contain a small quantity of mercury from previous test runs. Collect this mercury for later recycle. The cup can also be cleaned at this time by rinsing it with 0.1M nitric acid, wiping it with a KimWipe, and rinsing it with distilled water.

- 3) To a static teflon polarographic cell, add 10 to 15 ml of distilled water. Mount the cell in the polarograph using the spring-loaded arm to support the cell.
- 4) Disconnect the ribbon cable and flexible nitrogen supply tube from the rear of the Model 303A. Carefully remove the Model 303A from Module 1 and place it on a stable, level surface. If available, use of a ventilated fume hood is recommended.
- 5) Remove the ribbon cable (coded AA-JJ) connecting the Model 264A to Module 1. After unplugging its power cord, place the 264A next to the polarograph. Connect the two instruments using the coded AA-JJ ribbon cable. Connect the Model 264A to a 120 VAC power source.
- 6) Using the procedures outlined in the Model 303A manual, remove the glass capillary and pack for shipping. To prevent mercury leakage

during shipping, carefully drain the mercury from the polarograph (as described in the manual) and store in a leak-free container.

Using firm downward pressure, remove the glass reference electrode from the plastic support block and discard its liquid contents. To prevent the frit from drying out, place the electrode in a sealed plastic container filled with distilled water.

To minimize contamination during shipping and handling, it is recommended that sensitive components (such as the glass capillary, flow-through cell, and reference electrode) be placed in separate press-lock style plastic bags prior to their packing.

- 7) To remove the electrolyte reservoir from Module 1, first disconnect the flexible tygon tube from the filter column. Next loosen the teflon ferrule at the inlet of liquid pump LP1 and remove the teflon tube. Disconnect the level switch's electrical connector. Remove the reservoir from Module 1 and discard its contents in an appropriate manner.
- 8) Close the nitrogen regulator valve by turning it fully clockwise. Disconnect the flexible tubing at the top of the regulator and remove the nitrogen tank from Module 1.
- 9) Disconnect and remove the 1/4 inch tube which connects the Module 1 waste outlet to the polypropylene waste container. Dispose of the waste container's contents in an appropriate manner.
- 10) Disconnect all power cords and ribbon cables from the lead-in-air monitor's various components.
- 11) Remove the remaining components from the utility cart. Pack the components in a manner which will minimize the chance of their damage during shipping.

E.6 TROUBLESHOOTING GUIDE

<u>Symptom</u>	<u>Possible Cause and Solution</u>
Instrument fails "status check"	<p>Power cables or ribbon cables not plugged in or improperly connected. Check cables and connections.</p> <p>Component power switches not on. Check each instrument.</p> <p>Model 303A and Model 264A panel switches incorrectly set. Check Table E-2.</p> <p>Problem with electrical connectors. Check all connections.</p>
Computer program does not load.	<p>Disk in drive A. Make sure drive A is empty.</p> <p>Problem with main program. Load backup program from floppy drive.</p>
Instrument fails solution background check.	<p>Background lead content of water too high. Replace with better quality water.</p> <p>Electrolyte improperly prepared. Prepare fresh solution.</p>
Liquid leaks at fittings.	<p>Fitting improperly connected. Reattach tubing using outlined procedures. If problem continues, replace fitting and tubing.</p>

Polarographic cell overflows.

Liquid pump inoperative or out of calibration. Check behavior of pumps during main rinse cycle. Recalibrate or replace as necessary.

Insufficient nitrogen flowrate.

Nitrogen cylinder is empty. Check pressure and replace cylinder is below 50 psig.

Nitrogen solenoid inoperative. Check condition of solenoid and controlling electromechanical relay.

Rotameter improperly adjusted. Rotate valve clockwise to increase flowrate.

Regulator valve closed. Turn counter-clockwise to open valve.

Leak in tubing or connector. Inspect flow system for cause.

Copper peak not within $1.6V \pm 50\%$ during calibration check.

Electrolyte improperly prepared. Prepare fresh solution and repeat test.

Reference electrode solution has been diluted. Replace solution and repeat test.

Model 264A controls improperly set. Refer to Table E-2 for correct settings.

Response to 100 ppb lead standard not within 5%.

Lead standard improperly prepared. Prepare fresh standard and repeat test.

Reference electrode solution has been diluted. Replace solution and repeat test.

Model 264A controls improperly set. Refer to Table E-2 for correct settings.

"Low Electrolyte" message when reservoir is full.

Level switch connector not plugged in. Check connection.

Float switch stuck in open position. Inspect switch and replace if necessary.

Problem with electrical connection. Trace connection and check for continuity.

Air pump does not activate.

Ribbon cable not connected to Module 2. Check cable and connections.

Module 2 power cord not plugged in. Check cable.

Sampling less than 90% isokinetic.

Flow pressure drop too high. Close pump recirculation valve one turn and repeat test. If problem persists, replace cartridge filter.

Mass flow controller inoperative. Check flow controller and power supply.

Air pump malfunction. Check pump condition.

Sampling greater than 110% isokinetic.

Mass flow controller inoperative. Check flow controller and power supply.

Power surge occurs during test.

Load to power source is too high. Locate alternative outlet.

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Robert William Vanderpool was born on November 13, 1955, in Evanston, Illinois. He attended primary and secondary school in Ocala, Florida. In 1988, he received a Bachelors of Engineering degree from the University of Florida. He was awarded a Masters of Engineering (with thesis option) from the University of Florida in 1983. Following a six year period as an engineering consultant, he began a doctorate program at the University of Florida with a specialty in aerosol science and air pollution control.

I certify that I have read this study and that in my opinion it conforms to acceptable standards of scholarly presentation and is fully adequate, in scope and quality, as a dissertation for the degree of Doctor of Philosophy.



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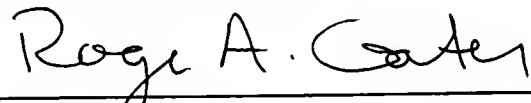
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